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U.S. Department of Energy  
Idaho Operations Office

***Laboratory Study Work Plan for Chemical  
Oxidation and Stabilization of V-Tanks'  
Contents, TSF-09/18, at Waste Area Group 1,  
Operable Unit 1-10***



Idaho National Engineering and Environmental Laboratory

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Prepared for the  
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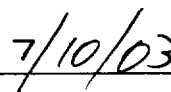
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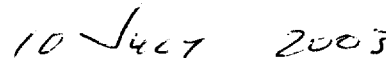
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## **ABSTRACT**

This Laboratory Study Work Plan describes objectives, methods, and materials needed to determine key preliminary design and operating parameters for treating and disposing of the V-Tanks' contents at Test Area North at the Idaho National Engineering and Environmental Laboratory.

This report provides information required to design and operate the selected ex situ chemical oxidation/stabilization process for remediation of the V-Tanks' contents. In addition, this report describes studies that will use both surrogates and actual V-Tank waste samples. The studies are structured to allow enough flexibility so that the results of initial tests will guide the direction of subsequent tests.



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## ACRONYMS

ANS	American Nuclear Society
ASTM	American Society for Testing and Materials
BBWI	Bechtel BXWT Idaho, LLC
BEHP	bis (2-ethylhexyl) phthalate
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
DOE	U.S. Department of Energy
DOE-ID	U.S. Department of Energy Idaho Operations Office
DRE	destruction and removal efficiency
EDF	Engineering Design File
EPA	U.S. Environmental Protection Agency
ES-CO/S	ex situ chemical oxidation followed by stabilization
FY	fiscal year
ICDF	INEEL CERCLA Disposal Facility
INEEL	Idaho National Engineering and Environmental Laboratory
INEL	Idaho National Engineering Laboratory
LRC	Lynchburg Research Center
M&O	management and operating
MCP	management control procedure
MLLW	mixed low-level waste
N/A	not applicable
PCB	polychlorinated biphenyl
PCE	tetrachloroethylene
PLN	plan
ppm	parts per million

PRD	program requirements document
RCRA	Resource Conservation and Recovery Act
SAP	Sampling and Analysis Plan
scfm	standard cubic feet per minute
SO	system operability
SOW	Statement of Work
SVOC	semivolatile organic compound
TAN	Test Area North
TBD	to be determined
TCA	trichloroethane
TCE	trichloroethene
TCLP	toxicity characteristic leaching procedure
TOC	total organic carbon
TSF	Technical Support Facility
USC	United States Code
UTS	universal treatment standard
VOC	volatile organic compound
WAG	waste area group

# **Laboratory Study Work Plan for Chemical Oxidation and Stabilization of the V-Tanks' Contents, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10**

## **1. PROJECT DESCRIPTION**

### **1.1 Basis for Preliminary Design and Laboratory Study Work Plan**

This Laboratory Study Work Plan describes objectives, methods, and materials for laboratory studies and mockup tests to determine preliminary design and operational parameters for treating and disposing of contents at the Idaho National Engineering and Environmental Laboratory (INEEL), Test Area North V-Tanks. The Test Area North (TAN), which is designated as Waste Area Group (WAG) 1, is one of 10 Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) waste area groups at the INEEL. Each of the four V-Tanks—designated as Tanks V-1, V-2, V-3, and V-9—contains a combination of liquid and sludge that is contaminated with metals, organics, and radionuclides. The preferred treatment alternative, upon which this laboratory study is based, is designated as ex situ chemical oxidation followed by stabilization (ES-CO/S). This alternative was identified from a list of seven candidate options that were considered. The flow sheets for all of the considered options and the selection process are documented in the *Technical Evaluation Report for the V-Tanks, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10* (DOE-ID 2003a).

A Record of Decision amendment will be finalized in calendar year 2003. If approved, this document will state that ES-CO/S will be the remedy for the V-Tanks' contents, with the INEEL CERCLA Disposal Facility (ICDF) identified as the targeted disposal site. The remedy is still proposed and is pending public/state input. Completion of the remedial action is expected by the year 2006. The purpose of this report is to determine the performance of ES-CO/S on the V-Tanks' contents and to provide information for further design of the treatment process. The technology is proven for aqueous systems, but it is not as proven for multiphase systems (such as the V-Tanks' contents). Parameters—such as pH, temperature, and mixing—need to be investigated.

This report has been prepared in accordance with the *Guide for Conducting Treatability Studies under CERCLA* (EPA 1989). In addition, this report provides technical and programmatic information for conducting CERCLA laboratory-scale treatability studies and full-scale cold mockup tests, and it provides details regarding the interface between the INEEL and the subcontracted facilities.

This report was identified in the Technology Evaluation Report (DOE-ID 2003a) as a secondary document titled *Preliminary Design Study Work Plan*. The title has been modified to be more representative of the work plan scope.

### **1.2 Description of the V-Tank Waste**

The V-Tanks are four underground tanks installed at the TAN Technical Support Facility (TSF) in the early 1950s as part of a system designed to collect and treat radioactive liquid effluents. The purpose of Tanks V-1, V-2, and V-3 (designated as TSF-09) was to store TAN-generated liquid radioactive waste before it was treated. Tank V-9 (designated as TSF-18) is a smaller, vertically oriented tank with a conical bottom; its purpose was to act as a clarifier through gravitation, and utilization of its designed shape, for the liquid streams entering Tanks V-1, V-2, or V-3. Waste was pumped into these tanks from the TSF laboratories and craft shops, the hot and warm shops, a radioactive decontamination shop, hot cells, and the

Initial Engine Test Facility. In 1968, a large quantity of oil (probably hydraulic oil) containing polychlorinated biphenyls (PCBs) at 680 parts per million (ppm) was discovered in Tank V-2. As a result, it was taken out of service. The oil was removed from Tank V-2 in 1981. The tanks have not been used since the 1980s, though liquid was accidentally discharged into Tank V-3 in the late 1980s. The V-Tank contents were sampled in 1993 and again in 1996.

Because the TSF-18 site (Tank V-9) is contiguous with and received the same waste as TSF-09 (Tanks V-1, V-2, and V-3), the two sites have been combined for the purpose of characterization and remedial analysis (ITC 1994). Total volumes of the tank contents are estimated at 1,880 gal of sludge and 10,032 gal of liquid. Table 1 provides the breakdown of each tank's waste volume.

Table 1. Estimated breakdown of V-Tank waste volume by tank.

Tank Identification	Capacity (gal)	Liquid Volume (gal)	Sludge Volume (gal)	Total Volume (gal)
V-1	10,000	1,164	520	1,684
V-2	10,000	1,138	458	1,596
V-3	10,000	7,660	652	8,312
V-9	400	70	250	320
Total	30,400	10,032	1,880	11,912

### 1.3 Description of Past V-Tank Contents' Data Collection

#### 1.3.1 Historical Sampling Campaigns

The data of interest span different sampling campaigns. A brief discussion of these campaigns follows. The V-Tanks were operated in the configuration shown in Figure 1.

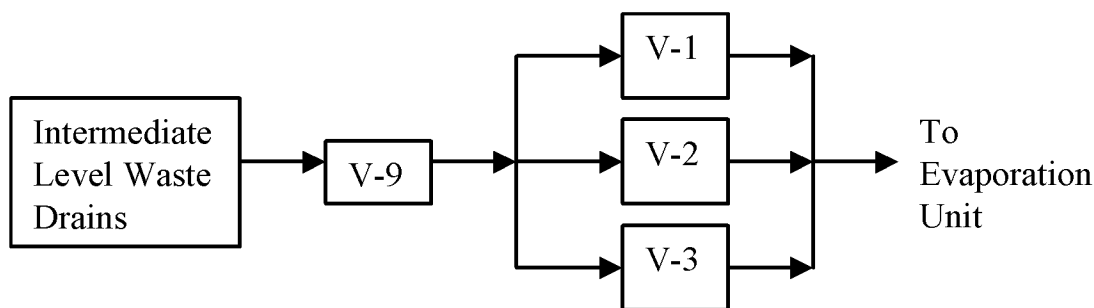


Figure 1. In-use block-flow configuration for the V-Tanks.

As depicted in Figure 1, waste from operations at TAN-607, TAN-633, and the Initial Engine Test Facility was pumped into Tank V-9 (acting as a trap for solids) before disposition into Tank V-1, V-2, or V-3. Liquid was removed from these tanks and was sent to an evaporator (the TAN-616 evaporator, the PM-2A evaporator, or the Idaho Chemical Processing Plant evaporator). This activity began in the mid-1950s. Tanks V-1 and V-3 remained in service until 1985.

Over time, solids accumulated in these tanks. In 1980, a sampling effort was undertaken to determine the composition of solids in the tanks. The solids were dried, and x-ray diffraction analysis was performed. The following narrative describes the sampling (Michael 1996):

In 1980, the tank still bottoms in all three tanks and the oil and water in Tank V-2 were sampled. One of the objectives of the sampling was to obtain full-depth core sediment samples to look for indications of stratification in the still bottoms. To accomplish this, a sample probe and container consisting of a 22-in. tube with a 22-ft-long detachable hollow aluminum handle was constructed. The sample tube was made out of 1/2 in. Schedule 40 polyvinyl chloride pipe, with a spring-loaded closure plate on the bottom. A polyvinyl chloride pipe cap was used to seal the top of the tube after it was detached from the handle. When the tanks were sampled, tanks V-1 and V-3 were still in use. Tank V-2 had a layer of oil on the liquid, which prevented the liquid wastes in the tank from being removed and processed. The tank was full, but not in use at the time of sampling. Two samples of the sediment were collected from each of the tanks through the 6-in. spare connection (located in approximately the center of the tank). The samples were a thin, black mud. Based on x-ray diffraction analysis and optical examination, the major components of the samples were silicone dioxide (sand, quartz, etc.), with most of the particles less than 10 µm in size. The analyst reported the presence of resin beads and ceramic-like particles (500 to 1000 µm) in all of the samples as well *[sic]*.

Table 2 shows the results of this sampling effort. The metal analysis was performed on dry solid and is reported on a dry basis.

After the sampling event in 1980, the oil was removed from Tank V-2 in 1981, and water was removed from the three tanks in 1982. Water continued to be removed from Tanks V-1 and V-3 from 1982 through 1985 and sent to an evaporation unit. No further additions or removals (i.e., evaporation) have occurred since 1985.<sup>a</sup>

In 1990, two separate liquid-sampling campaigns were conducted, one for Tanks V-1 and V-2 and the other for Tank V-3. There is no available information on the method used for collecting the samples, though it is suspected they were collected through the 3-in. or 6-in. connections. All of the analyses were performed on-Site for Resource Conservation and Recovery Act (RCRA) hazardous constituents, gamma constituents, and gross alpha/beta. A decision was made to use the 1993/1996 data, because the 1990 data only had analysis for RCRA-characteristic metals, volatile organic compounds (VOCs), and semivolatile organic compounds (SVOCs) (eight metals, 10 VOCs, and 14 SVOCs), while the other campaigns analyzed for these plus many more analytes.

In 1993, as part of the Operable Unit 1-05 Track 2 investigation, another sampling campaign was performed. Liquid samples were collected from unknown depths in Tanks V-1, V-2, and V-3, using a peristaltic pump and Teflon tubing fastened to a stainless-steel pole and inserted into the tank through the open manhole. The sediment samples were collected from the bottom of each tank using a 12-ft Teflon composite liquid-waste sampler (i.e., COLIWASA). Both types of samples were analyzed for VOCs, metals, gamma radionuclides, uranium, thorium, plutonium and tritium isotopes, total strontium, gross beta, and miscellaneous information (i.e., anions, pH, and percent dissolved solids). Results of this sampling activity are included in Appendix A of the *Comprehensive Remedial Investigation/Feasibility Study for the Test Area North Operable Unit 1-10 at the Idaho National Engineering and Environmental Laboratory* (DOE-ID 1997).

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a. There has been some in-leakage of water into the V-Tanks, in particular Tank V-3. This water represents the groundwater from rain and snowmelt that infiltrated into the tank.

Table 2. Breakdown of V-Tank waste data by tank from the 1980 sampling effort.

Waste Characteristics	Tank V-1	Tank V-2	Tank V-3
pH	8.6	9.53	9.57
Undissolved solids (g/L)	136	53	81
Density (g/ml)	0.9	1.1	1.1
Bulk Metals (Based on X-Ray Diffraction)	Concentrations <sup>a</sup> of Bulk Metals Tank V-1 (mg/kg)	Concentrations <sup>a</sup> of Bulk Metals Tank V-2 (mg/kg)	Concentrations <sup>a</sup> of Bulk Metals Tank V-3 (mg/kg)
Ag	20	50	60
Al	6,000	10,000	8,000
Bo	800	1,000	400
Be	400	300	400
Ca	20,000	20,000	20,000
Cr	6,000	10,000	800
Cu	20	30	8,000
Fe	30,000	50,000	50,000
Mg	30,000	20,000	30,000
Mn	8,000	2,000	10,000
Na	1,000	600	1,000
Ni	500	1,000	800
Pb	200	200	200
Si	240,000	200,000	190,000
Sn	70	100	80
Zn	2,000	2,000	4,000
P	110,000	120,000	130,000
Ba	1,000	4,000	700

a. Concentrations are based on the dry solids in the V-Tanks.

In 1996, another sampling campaign began. This campaign was performed to fill some existing data gaps (such as obtaining values for SVOCs and PCBs). In addition to Tanks V-1, V-2, and V-3, sampling was performed on Tank V-9 to obtain information on its contents. Results of this sampling activity are included in Appendix A of the Remedial Investigation/Feasibility Study (DOE-ID 1997).

Each sampling campaign had unique, specific objectives. Because of these differences, no one data set should be used exclusively to characterize the waste. A combination of all the existing data should be used for Tanks V-1, V-2, and V-3 (see Table 3). Data for Tank V-9 come from the 1996 campaign. The following sections discuss the data in detail.

Table 3. Data used to characterize Tank V-1, V-2, and V-3 waste based on 1993 and 1996 sampling campaigns.

Analysis	Liquid	Sludge
Bulk metals	1993 data	1980 data <sup>a</sup>
RCRA metals	1993 data	1996 data <sup>b</sup>
Radionuclides	1996 data	1996 data <sup>b</sup>
Volatile organic compounds	1993 data	1993 data <sup>b</sup>
Polychlorinated biphenyls	1996 data	1996 data <sup>a</sup>
Semivolatile organic compounds	1996 data	1996 data <sup>a</sup>
Miscellaneous: anions, pH, total C	1996 data	1996 data <sup>b</sup>
Percent of solid in sludge	N/A	1993 data <sup>c</sup>

a. Data are reported on a dry-weight basis.

b. Data are reported on a wet-weight basis.

c. The percent of solids in the sludge is based on 1993 data.

N/A = not applicable

RCRA = Resource Conservation and Recovery Act

### 1.3.2 Bulk and Resource Conservation and Recovery Act Metals' Data

In 1980, the objective of the sampling and analysis effort was to identify the nature of the solids that resided at the bottom of the V-Tanks.<sup>b</sup> The x-ray diffraction was used to identify bulk metal concentrations within the dried solids. The concentrations were reported on a dry basis.<sup>e</sup> The sludge-phase analyses from the 1993 and 1996 sampling campaigns were performed on decanted sludge in 1993 and on gravity-filtered sludge in 1996. An acid digestion method used on both types of sludge to determine metal concentrations was followed by either inductively coupled plasma atomic-emission spectroscopy or flame atomic-adsorption spectroscopy. The results were reported on a wet mass without moisture correction.<sup>d</sup>

The main difference in the metals' data set is that by using an acid digestion method, the 1993 and 1996 metal data results showed very little silicon. The 1980 metal data results report a silicon concentration in the dried solid of 20 to 22%. The acid digestion method is adequate for determining concentrations for most metals (such as RCRA metals), but fails for metals that form highly insoluble species (such as SiO<sub>2</sub> or Al<sub>2</sub>O<sub>3</sub>). For data decisions related to metal analysis, the consensus was to use the bulk metals' data results from 1980 for metals with a concentration greater than 0.5% in the dried solids and the 1996 data results for RCRA metals (as characteristic and as underlying hazardous constituents). For metals' characterization in the aqueous phase, data results from 1993 were used.<sup>e</sup>

### 1.3.3 Radiological Data

The radiological data used for these studies are from the 1996 sampling and analysis campaign. A complete nuclide analysis set was performed in that year to examine the V-Tank waste. The sludge-phase results were reported as wet mass without moisture correction.

b. The RCRA guidelines were only beginning to become known by this time; thus, hazard identification was not performed.

c. Analysis for metals' concentration was the only one performed in the 1980 sampling effort.

d. In 1993, the sludge sample was settled and the aqueous layer was decanted away. In 1996, because of the highly disturbed sludge resulting from the collection method, the samples had to be gravity-filtered to produce similar moisture content of the sludge as that of the 1993 sampling campaign.

e. No metals' analysis for the aqueous phases was performed for Tanks V-1, V-2, and V-3 in 1996.

#### **1.3.4 Volatile Organic Compound Data**

Volatile organic compound data were obtained for Tanks V-1, V-2, and V-3 from both the 1993 and 1996 campaigns, and only from Tank V-9 in the 1996 campaign. It was determined from conversations with sampling personnel that the 1993 sludge data for VOCs were more reliable because of the sampling method employed. As a result, the VOC data from 1993 were used for Tanks V-1, V-2, and V-3. The VOC data from 1996 were used for Tank V-9. All of the sludge-phase VOC data were reported on a wet basis without moisture correction.

#### **1.3.5 Semivolatile Organic Compound Data**

The SVOC results for all of the four tanks are from the 1996 sampling and analysis campaign. The SVOC sludge-phase results were reported on a dry basis with moisture correction.

#### **1.3.6 Polychlorinated Biphenyl Data**

The PCB results for all four tanks are from the 1996 sampling and analysis campaign. The PCB sludge-phase results were reported on a dry basis with a moisture correction. The congeners that were analyzed are Aroclor-1016, Aroclor-1221, Aroclor-1232, Aroclor-1242, Aroclor-1248, Aroclor-1254, and Aroclor-1260. Only Aroclor-1260 was detected, which is typically found only in the sludge phase.

### **1.4 Contaminants to be Addressed in the Preliminary Design and Laboratory Studies**

The contents of the combined liquid and sludge found in the V-Tanks are summarized in Table 4. The contents in both V-Tanks are hazardous and radioactive. Because of the radioactivity level, treatment operations need to be performed remotely. Surrogate feed stocks will be used in the preliminary laboratory testing due to the extensive testing planned. The surrogate feedstock will allow the treatment procedure to be screened and refined. Once completed, actual tank waste will be used for testing.

The following contaminants for treatment are examined in this report:

- Trichloroethylene
- Tetrachloroethylene
- 1,1,1-Trichloroethane
- PCBs (Aroclor-1260)
- Cadmium
- Chromium
- Lead
- Mercury
- Bis (2-ethylhexyl) phthalate.

Chemical oxidation will treat the organic contaminants and stabilization will treat the metal contaminants.

Table 4. Concentrations of key elements and compounds found in V-Tank waste contents.<sup>a</sup>

Key Elements and Compounds	Tank V-1 Waste Contents (mg/kg)	Tank V-2 Waste Contents (mg/kg)	Tank V-3 Waste Contents (mg/kg)	Tank V-9 Waste Contents (mg/kg)	Average Waste Contents from Tanks V-1, V-2, V-3, and V-9 (mg/kg)
<b>Inorganics</b>					
Aluminum (Al)	527	1,120	923	2,690	967
Antimony (Sb)	5.13	5.35	0.958	11.5	2.44
Arsenic (As)	3.00	3.45	0.860	3.05	1.57
Barium (Ba)	43.3	38	11.5	299	27.9
Beryllium (Be)	8.31	4.24	1.49	20.2	3.36
Cadmium (Cd)	20.2	22.7	5.09	21.8	10.1
Calcium (Ca)	1,780	2,240	2,340	6,750	2,420
Chloride (Cl)	208	102	59.9	397	96.1
Chromium (Cr)	526	1,120	25.8	1,880	296
Iron (Fe)	2,630	5,580	5,770	14,600	5,350
Lead (Pb)	255	303	72.6	454	140
Magnesium (Mg)	2,640	2,240	3,470	9,010	3,230
Manganese (Mn)	702	2,230	1,150	4,270	1,500
Mercury (Hg)	205	116	51.7	1,670	129
Nickel (Ni)	81.4	76	23.8	319	47.5
Phosphorus (P)	9,630	13,400	15,000	40,400	14,500
Silicon (Si)	21,000	22,300	21,900	70,700	24,600
Silver (Ag)	35.2	50.5	6.95	522	31.7
Zinc (Zn)	4,460	417	1,340	1,410	1,980
<b>Volatile organic compounds</b>					
Tetrachloroethylene	438	138	36.3	425	118
Trichloroethane	0.314	0.156	0.044	1,770	51.4
Trichloroethene	3.85	0.362	0.216	14,500	421
<b>Semivolatile organic compounds</b>					
Bis (2-ethylhexyl) phthalate	919	586	338.0	345	453
<b>Polychlorinated biphenyls</b>					
(Aroclor-1260)	34.6	24.4	10.1	95.9	17.9
<b>Radionuclides</b>					
Cesium-137	nCi/g	nCi/g	nCi/g	nCi/g	nCi/g
	1,740	1,810	527	4,480	984
Strontium-90	1,520	3,200	1,500	5,180	1,840
Transuranic waste <sup>b</sup>	11	4.02	2.04	26.4	4.27

a. The values are for the combined liquid and sludge phases of the V-Tank waste.

b. The transuranic constituents include Pu-238, Pu-239/240, Am-241, Cm-243/244, and Np-237.

## 1.5 Test Description

The CERCLA guidelines have identified three main steps in the tiered approach for treatment testing: (1) laboratory screening, (2) bench-scale testing, and (3) full-scale testing (EPA 1989). This work plan study discusses a combination of all three. The study will consist of four test phases, which are further described in Section 1.6.

The bench-scale testing will consist of two separate studies. These studies consist of the following:

- Testing with surrogate samples (i.e., cold study)
- Testing with V-Tank waste samples (i.e., hot study).

The cold study will investigate the operation parameters that influence chemical oxidation and stabilization, which involve the following:

- Defining the effective stoichiometry and kinetics for specific oxidant systems and the V-Tank surrogate
- Defining the off-gas generation rate and composition at given oxidation temperatures
- Selecting a set of conditions that allow for effective chemical oxidation of the V-Tank surrogate
- Selecting stabilization additives and mixing schemes that allow for effective stabilization of an oxidized surrogate.

The hot study will be performed after the cold study. Samples of actual V-Tank waste will be treated with the optimal parameters, as determined from the cold study. Where feasible, the exact same equipment used for the cold study will be used for the hot study.

In addition to the two bench studies, there will be a full-scale cold mockup test. This test will be performed with either the same surrogate as the cold laboratory study or a modification of the surrogate.<sup>f</sup> The purpose of the full-scale cold mockup test is to determine if there are any significant process problems due to scale-up from the laboratory to the actual equipment configuration. The full-scale test will eventually lead to a system operability (SO) test before moving the equipment to TAN. Potential problems could include the following:

- Mixing of multiphase V-Tank waste with oxidants
- Heat effects from oxidation reactions
- Foaming effects during oxidation process.

This testing is to verify that the selected V-Tank remedy will meet the treatment criteria and be ready for implementation in Fiscal Year (FY) 2005.

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f. Depending on the specific piece of information that is needed from a full-scale study, the makeup of the surrogate might be constrained by time or cost. Any modification should still be reflective of a critical aspect of the actual V-Tank waste.

## **1.6 Test Phases of the Laboratory Study Work Plan**

To satisfy data quality objectives, the Laboratory Study Work Plan will be divided into four test phases. Bench-scale cold tests will be performed in Test Phase 1 (screening tests) and Test Phase 2 (verification tests). Test Phase 3 will comprise bench-scale hot tests, and Test Phase 4 will comprise full-scale cold mockup tests. Screening tests produce data that are taken for preliminary selection or preliminary adjustment of mix parameters. The work plan activities include the following:

1. Bench-scale cold tests (Test Phases 1 and 2)
  - a. Test plan
    - (1) Selection of surrogate
    - (2) Description of testing
  - b. Cold laboratory tests (screening and verification)
  - c. Final report
2. Bench-scale hot tests (Test Phase 3)
  - a. Test plan
    - (1) Providing samples from archive
    - (2) Shipping samples to laboratory
    - (3) Description of testing (same as bench-scale cold testing)
  - b. Hot laboratory tests
  - c. Final report
3. Full-scale cold mockup tests (Test Phase 4)
  - a. Test plan
  - b. Cold mockup tests
  - c. SO test
  - d. Final report.

The results of the cold bench-scale testing (Test Phases 1 and 2) will feed into both the hot bench-scale testing and the full-scale cold mockup testing (Test Phases 3 and 4).

## 2. DESCRIPTION OF REMEDIAL TECHNOLOGIES

The ES-CO/S is a low-temperature process that uses chemical reactions in the aqueous phase to destroy organic constituents, followed by stabilization to immobilize remaining metals to meet land disposal restrictions in accordance with the requirements of 40 *Code of Federal Regulations* (CFR) 268, “Land Disposal Restrictions.” Typical feed streams for chemical oxidation consist of aqueous streams contaminated with organics. In deciding between using chemical oxidation versus chemical reduction as the main reaction scheme—both of which serve to remove chlorinated hydrocarbons from an aqueous waste stream—chemical oxidation was chosen due to:

- The presence of bis (2-ethylhexyl) phthalate, which requires oxidative attack of the carbon chains
- The presence of a significant amount of oil and grease, which might require oxidative destruction for oil/grease breakdown in order to, if necessary, facilitate destruction of contaminant organics.

Feed streams from the V-Tanks would not be considered typical. The V-Tank waste—taken as a composite from Tanks V-1, V-2, V-3, and V-9—is a settled slurry (i.e., liquid and sludge phase), where the combined liquid and sludge phase together contain approximately 6 wt% solids. Most of the contaminants for treatment reside in the sludge phase (i.e., a mixture of particulates, oil, and water). The particle size distribution of solids in Tank V-9 is given in Table 5.

Table 5. Particle size distribution for Tank V-9 sludge.

Sieve Size	Sample Number 2CB90301 (wt%)	Sample Number 2CB90302 (wt%)
+30 mesh (greater than 600 $\mu\text{m}$ )	65.8	70.0
-30 + 50 mesh (between 300 and 600 $\mu\text{m}$ )	8.4	7.9
-50 + 70 mesh (between 212 and 300 $\mu\text{m}$ )	1.7	1.8
-70 + 100 mesh (between 150 and 212 $\mu\text{m}$ )	3.4	3.5
-100 + 200 mesh (between 75 and 150 $\mu\text{m}$ )	4.4	5.0
-200 + 400 mesh (between 38 and 75 $\mu\text{m}$ )	3.5	3.1
-400 mesh (less than 38 $\mu\text{m}$ )	12.7	8.7

Since the purpose of Tank V-9 was to act as a solid liquid separator, it is assumed that the particles in Tanks V-1, V-2, and V-3 are likely to be smaller and less dense than the particles residing in Tank V-9.

### 2.1 Chemical Oxidation Process

The chemical oxidation process involves the chemical destruction of the organic contaminants. This destruction is facilitated by an oxidant that decomposes into highly reactive free radicals within the aqueous solution. Input heat or the presence of catalytic species accelerates the decomposition of oxidant into these free radicals. The targeted organics of concern in the V-Tanks that require destruction are tetrachloroethylene, trichloroethylene, 1,1,1-trichloroethane, Aroclor-1260, and bis (2-ethylhexyl) phthalate. The intention of chemical oxidation is to destroy the chlorinated hydrocarbons to produce carbon dioxide, water, and hydrogen chloride. There is also the potential to create intermediate products by incomplete degradation. Intermediates potentially formed from the targeted V-Tank organics include, but are not limited to, the following: 1,1-dichloroethylene, 1,2-dichloroethylene (cis or trans), vinyl chloride, biphenyl, benzene, and phthalic acid.

Oxidants that are considered in this study include sodium persulfate and Fenton's reagent (hydrogen peroxide and ferrous salt). Each oxidant system works within a preferred pH regime. Organic contaminant destruction is based on two main pathways: (1) breakage of a carbon-carbon bond (pi and/or sigma) or (2) replacement of a halogen (chloride) group from the molecule. In theory, if the reaction goes to completion, the overall chemical reaction (unbalanced) would be as follows<sup>g</sup>:



The general equation is the same for both oxidant systems, except that sodium and  $\text{SO}_4^{2-}/\text{HSO}_4^-$  will result from the addition of sodium persulfate oxidant. The destruction efficiencies required for the V-Tanks, taken as a single waste stream to bring the organic concentration below the universal treatment standards (UTSs) in the postoxidation slurry, are presented in Table 6.

Table 6. Maximum organic-destruction efficiencies for V-Tanks taken as a single waste.

Organic Contaminant	Total Concentration <sup>a</sup> (mg/kg)	Universal Treatment Standard (mg/kg)	Destruction Efficiency (as is %)	Destruction Efficiency without 6,000 gal <sup>b</sup> (%)
Tetrachloroethylene	118	6	94.9%	97.5
Trichloroethylene	421	6	98.6%	99.3
1,1,1-Trichloroethane	51.4	6	88.3%	94.3
Aroclor-1260	17.9	10	44.1%	72.1
Bis (2-ethylhexyl) phthalate	453	28	93.8%	96.9

a. The total concentration is based on the contents of all the tanks averaged. Depending on the exact nature of a chosen batch, these required destruction efficiencies could be different.

b. This column represents the required destruction efficiencies after the removal of 6,000 gal of liquid supernatant from Tank V-3.

The destruction efficiencies presented in Table 6 assume the following<sup>h</sup>:

- The waste stream is a composite of all the V-Tanks' contents.
- No credit is taken for the mass of oxidant additives needed.
- No credit is taken for the mass of additives needed to produce the final, cemented waste form.
- The Aroclor-1260 regulatory limit is for the summation of all aroclors, so the destruction efficiency implies that intermediates are not a PCB.
- No credit is taken for volatility and escape of VOCs from the waste mixture. Heating of the reaction mixture could result in the loss of VOCs because of their volatility. As a result, the

g. The stoichiometric ratio between oxidant and organic is based on total organic carbon. For two candidate oxidant systems (peroxydisulfate and hydrogen peroxide), the stoichiometric molar ratio of oxidant to organic carbon is 3.

h. These destruction efficiencies represent the worst-case. Credit for oxidant weight increase and stabilization additives will be used to establish achievement of final UTS values.

necessary destruction of a VOC by chemical oxidation might be reduced, based on the amount of the VOC that passes through the recycle condenser and is collected in the off-gas system.

For actual treatment, the organic treatment standards presented in Table 6 would be based on total concentration in the final waste form, and in the case of the V-Tank contents, this would be the concentration in the final, solidified waste form.

## 2.2 Chemical Oxidation Parameters

The oxidant choices to be studied are sodium persulfate and Fenton's reagent (hydrogen peroxide and soluble ferrous salt). Both oxidants rely on an initiation reaction to form free radicals as expressed in Equations (2) and (3):



It is anticipated that either of these free radicals could work on the V-Tank waste. In addition to using a single oxidant to treat the contaminants and the resident oily phase, a combination of the two oxidants can be used.

Temperature and pH affect the kinetics for organic destruction using these oxidant systems. The hydrogen peroxide (Fenton's reagent) system works best in a pH range of 3 to 5. The persulfate system has kinetics that are independent at pH>1. The rate of reaction for hydrogen peroxide destruction of organics is expected to be adequate at temperatures as low as 40°C, whereas the persulfate will require the system temperature to be as high as 80 to 90°C. Since the V-Tank waste contains volatile contaminants, such as trichloroethane (TCA) and trichloroethene (TCE), these elevated temperatures could volatilize significant portions of VOCs from the oxidant bath.

Hydrogen peroxide, when used in Fenton's reagent, requires a catalyst to assist the oxidation process, namely the ferrous ion. This introduces another parameter to control, that being the ratio of iron to hydrogen peroxide. The persulfate system does not require a catalyst, though heat is required to initiate radical formation. Much of the organic contaminant resides in the sludge phase, which consists of roughly 60 to 70% water, 20 to 30% inorganic solid, with the remainder being organic (i.e., oil and grease). Within this sludge phase, the organic contamination is believed to reside mostly in the oil phase, with a small portion adsorbed onto the solids. Since the free radical will be present in the aqueous phase, organic destruction must occur in the aqueous phase. In addition, some oxidation could occur at the interface of the aqueous phase and either the oil phase or solid phase. Fairly high shear mixing should emulsify the phases to the extent that free radical attack can be effective. The energy input to the system from mixing needs to be quantified, and an optimal mixing protocol (i.e., blade configuration and rotational speed) needs to be determined.

The amount of oxidant is another parameter to consider. Using V-Tank data, the quantity of organic carbon can be estimated. Assuming the carbon is primarily in long, straight chain alkanes (i.e., hydraulic or cutting oils), the idealized stoichiometric mole ratio of oxidant to carbon is 3 to 1 for either oxidant system. The addition of oxidant will be varied as a factor (greater than unity) of this stoichiometric ratio. In addition to the amount of oxidant, the addition rate of the oxidant will be a parameter of interest. Free radical reactions are exothermic and autocatalytic; therefore, runaway reactions are a concern if too much oxidant is added at once.

Off-gas primarily consisting of water vapor, VOCs, and carbon dioxide will be generated from the process. The amount of water vapor and VOCs in the off-gas is related to the oxidation temperature. The carbon dioxide in the off-gas is related to the destruction of organics. Other constituents that could be in the off-gas at low levels are carbon monoxide, hydrogen chloride, and mercuric chloride, among others.

Another aspect of interest in the chemical oxidation process is the relative susceptibility of various organic constituents to free radical attack. The V-Tanks contain various types of organics such as cross-linked polymers (i.e., resin beads), long-chained alkanes (i.e., mineral oil), phenyls (i.e., PCBs and phthalates), and chlorinated alkanes/alkenes (i.e., TCA and TCE).

## 2.3 Stabilization Step

Once the oxidation of the waste has been completed, the resulting mixture needs to be stabilized to:

- Create a solid waste form without free liquids
- Immobilize RCRA metals (i.e., Pb, Hg, and Cd) to allow the waste form to meet the UTSS when subjected to the toxicity characteristic leaching procedure (TCLP).

The stabilization agent will be cement-based with certain additives chosen to immobilize RCRA metals (such as mercury). In addition to complying with RCRA treatment standards for metals, stabilization provides the added benefit of immobilizing radionuclides. The stabilization step requires that the postoxidant slurry have a basic pH. The slurry will require a pH adjustment from acidic to basic. Sodium hydroxide can be used to raise the slurry. The stabilizing agents that will be considered are three different types of Portland cement with varying additives (such as blast furnace slag, silica fume, and fly ash). The *Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of Surrogate V-Tank Waste (TSF-09/18) at Waste Area Group 1, Operable Unit 1-10* (INEEL 2003a) for Test Phases 1 and 2 discusses these recipes in more detail. The stabilization agent and oxidized waste need to be well mixed to ensure that the waste form is as homogeneous as feasible, as it cures in suitable storage/disposal containers.

### 3. SPECIFIC TEST OBJECTIVES

This report provides information required to evaluate the chemical oxidation and stabilization process for treatment of the V-Tanks' contents. The Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of Surrogate V-Tank Waste (INEEL 2003a) allows enough flexibility in the testing protocol so the results of initial tests can guide the direction of subsequent tests. It is not intended to perform an exhaustive study of all parameters for optimization of the remediation process. These tests must determine suitable and effective process conditions for remediation of the V-Tank waste.

Test Phases 1 and 2 (bench-scale cold tests) objectives are as follows:

- Determine effective chemical oxidation parameters and the ability to meet land disposal restrictions for VOCs and SVOCs by determining process:
  - Oxidant input amount and rate
  - Catalyst concentration and input rate to promote reaction without autocatalytic concerns
  - pH
  - Temperature
  - Mixing energy
  - Surrogate composition
  - Sequence and relative rates of destruction of organics
- Determine effective stabilization parameters and the ability to meet land disposal restrictions for metals by determining:
  - Stabilization agent recipe
  - Leachability of RCRA metals from the stabilized waste form
- Obtain feasibility data by:
  - Measuring heat generation rate
  - Measuring off-gas flow rate
  - Evaluating durability of construction materials
  - Measuring the partitioning of VOCs from the waste mixture to the off-gas.

Test Phase 3 (the bench-scale hot tests) objective is as follows:

- Determine the effectiveness of treatment on samples of actual V-Tank contents using the optimized/ preferred conditions determined from Test Phases 1 and 2.

Test Phase 4 (the full-scale cold mockup tests) objective is as follows:

- Determine the effectiveness of treatment with the equipment anticipated to be used in the field for actual V-Tank contents' removal (to the extent feasible) and treatment using preferred conditions, selected from those used in Test Phases 2 and 3.

Table 7 presents the specific test objectives and criteria. The analytical levels are reported as definitive or screening for the specific test criteria measures. When screening is reported, the measure is from Test Phase 1 testing.

These test phases are set up so that each test phase feeds information into the next test phase. As a result, these test phases (from 1 to 4) will be performed sequentially.

Table 7. Specific test objectives and criteria.

Objective	Test Criteria Measure	Data Usage	Proposed Method	Detection Level	Analytical Level
Determine effectiveness of chemical oxidation.	Initial characterization of surrogate containing key constituents desired to match actual V-Tank waste, consolidated for treatment (Test Phase 1 and 2)	Needed to establish initial conditions for chemical oxidation.	VOC: Method 8260B	See Note 1.	Definitive
			SVOC: Method 8270C	See Note 1.	
			PCB: Method 8082	See Note 1.	
			TCCLP: Method 1311	See Note 2.	
Determine effectiveness of stabilization agent.	Postoxidation concentration of VOCs and SVOCs (Test Phase 1)	Needed to select oxidant input sequence, catalyst concentration, pH, temperature, mixing energy, and amount of oxidant(s).	Metals: Method 3051, 6010		
			Anions: Method 300.0		
			TOC: Method 415.1		
			Subcontract laboratory method (TBD)	TBD	Screening
Determine effectiveness of stabilization agent.	Postoxidation concentration of VOCs and SVOCs (Test Phase 2)	Needed to quantify DREs and to ensure that VOCs/SVOCs are below UTS (for final waste form).	VOC: Method 8260B	Three to five times below UTS levels for TCA, TCE, PCE, BEHP, and PCB	Definitive
			SVOC: Method 8270C		
			PCB: Method 8082		
			TCCLP: Method 1311		
Determine effectiveness of stabilization agent.	Characterize the postoxidation surrogate slurry—concentrations of metals, sulfates, pH, viscosity, slurry bulk density, and re-suspension requirements. (Test Phase 1 and 2)	Needed to establish initial conditions for stabilization tests and to determine the change in the leachability of the metals because of oxidation.	Metals: Method 3051, 6010	See Note 1.	Definitive
			Anions: Method 300.0	See Note 1.	
			TOC: Method 415.1	See Note 1.	
			VOC: Method 8260B	See Note 2.	
Determine effectiveness of stabilization agent.	TCLP metals for stabilized sample (Test Phase 2)	Needed to establish that the stabilized waste form meets the UTS for disposal.	Method 1311, 3051, 6010 (see Note 2)	Order of magnitude below UTS limits for metals	Definitive
Determine effectiveness of stabilization agent.	Compressive strength	Needed to determine the strength of the final stabilized form.	ASTM C109-95	0.5 psi	Screening
			ASTM C39-94		
Determine effectiveness of stabilization agent.	Free water determination	Needed to ensure that the final stabilized form meets the ICDF waste acceptance criteria.	SW 846-9095		Definitive
			ANS 55.1 App. 2		

Table 7. (continued).

Objective	Test Criteria Measure	Data Usage	Proposed Method	Detection Level	Analytical Level
Determine effectiveness of chemical oxidation on actual V-Tank sample. (Test Phase 3)	Initial characterization of V-Tank sample from a feed recipe is provided by BBWL.	Needed to establish initial conditions for chemical oxidation and stabilization.	VOC: Method 8260B	See Note 1.	Definitive
			SVOC: Method 8270C	See Note 1.	
			PCB: Method 8082	See Note 1.	
			TCLP: Method 1311	See Note 2.	
Determine the feasibility of treatment. (Test Phases 1, 2, and 4)	Postoxidation concentration of VOCs and SVOCs	Needed to quantify DREs and to ensure that VOCs/SVOCs are below UTS (for final waste form).	Metals: Method 3051, 6010		
			Anions: Method 300.0		
			TOC: Method 415.1		
			VOC: Method 8260B	Three to five times below UTS levels for TCA, TCE, PCE, BEHP, and PCB.	Definitive
	TCLP metals for stabilized sample	Needed to establish that the stabilized waste form meets the UTS for disposal.	SVOC: Method 8270C		
			PCB: Method 8082		
	Compressive strength	Needed to determine the strength of the final stabilized form.	Method 1311, 3051, 6010 (see Note 2)	UTS limits for metals	Definitive
	Free water determination	Needed to ensure that the final stabilized form meets the ICDF waste acceptance criteria.	ASTM C109-95		Screening
			ASTM C39-94		
	Rate of heat generation	Needed to design the reactor's cooling/heating system.	SW 846-9095 ANS 55.1 App. 2		Definitive
Determine the feasibility of treatment. (Test Phases 1, 2, and 4)	Rate of heat generation	Needed to design the reactor's cooling/heating system.	Two type K thermocouples—(every 30 seconds)	20 to 100°C (± 0.5°C)	Screening
			Method TBD		
	Rate of oxidant addition	Needed to design the reactor's feed system.	Subcontract laboratory method (TBD)	TBD	Screening
	Rate of gas generation	Needed to design the reactor's off-gas system.	Flow meter	0.01 to 0.1 scfm	Screening
			Pressure transducer (closed system with relief valve)	TBD	Screening
	Anion generation in oxidation process	Needed to determine effects on the stability of grouted waste form.	Method 300.0	TBD	Definitive

Table 7. (continued).

Objective	Test Criteria Measure	Data Usage	Proposed Method	Detection Level	Analytical Level
	Rate of reactions	Needed to predict times required to reach the desired extent of reaction.	VOC: Method 8260B SVOC: Method 8270C PCB: Method 8082 Measurements are taken every 4 hours.	UTS levels for TCA, TCE, PCE, DEHP, and PCB	Screening
	Compatibility of materials with slurry during and after oxidation	Needed to determine if there are special requirements for piping and the stabilization unit.	Measure weight loss from coupons.	TBD	Screening
	Sequenced rate of organic destruction	Needed to determine if background oil needs to be destroyed in order to destroy organic contaminants.	TBD	TBD	Screening
	Percentage of VOCs that escape to the off-gas system	Needed to determine the composition of condensate and the composition of off-gas beyond the condenser.	VOC: Method 8260B SVOC: Method 8270C PCB: Method 8082 Liquid N <sub>2</sub> trap (do a mass balance)	TBD TBD TBD	Definitive

Note 1: In using the assigned methods, only the actual target spikes need quantification.

Note 2: The TCLP detection limits will be the UTS levels for metal species. Method 1311 describes the TCLP extraction; the proposed determinative methods would then be 3051 and 6010.

ANS = American Nuclear Society

ASTM = American Society for Testing and Materials

BBWI = Bechtel BWXT Idaho, LLC

BEHP = bis (2-ethylhexyl) phthalate

DRE = destruction and removal efficiency

ICDF = INEEL CERCLA Disposal Facility

PCB = polychlorinated biphenyl

PCE = tetrachloroethylene

sefm = standard cubic feet per minute

SVOC = semivolatile organic compound

TBD = to be determined

TCA = trichloroethane

TCE = trichloroethene

TCLP = toxicity characteristic leaching procedure

TOC = total organic carbon

UTS = universal treatment standard

VOC = volatile organic compound

## 4. EXPERIMENTAL DESIGN AND PROCEDURES

This section defines test parameters, levels of testing, the type and amount of replication, and the volumes of tank contents required for testing. The preliminary design and Laboratory Study Work Plan will be performed in four test phases:

- Test Phase 1—Bench-scale cold tests, screening of parameters
- Test Phase 2—Bench-scale cold tests, validation of accepted parameters
- Test Phase 3—Bench-scale hot tests, using accepted parameters
- Test Phase 4—Full-scale cold mockup tests.

Tests performed for each phase of testing are summarized below and will be described in detail in the appropriate test plans. Test Phase 1 data quality is defined as screening level (e.g., choosing one oxidant over another), while Test Phases 2 through 4 data quality is defined as definitive. It may be noted that although Phase 1 is considered as screening, the data quality could be elevated to definitive at the subcontractor's discretion.

### 4.1 Test Phases 1 and 2—Experimental Design and Procedures for Bench-Scale Cold Tests

The laboratory subcontractor, MSE Technology Applications, has prepared the Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of Surrogate V-Tank Waste (INEEL 2003a) for this work. A surrogate recipe is presented in the Cold Bench-Scale Test Plan according to a recipe that Bechtel BWXT Idaho, LLC (BBWI) formulated.

#### 4.1.1 Test Phase 1

The purpose of the screening tests is to examine aspects of chemical oxidation and stabilization. The following test objectives will be considered for this test phase:

##### Test Objective 1

To determine which of the potential process scenarios are effective in treating surrogate V-Tank waste to regulatory compliance levels and to identify suitable and effective process conditions for the chemical oxidation process scenario for future full-scale cold mockup testing and field deployment. Initial scoping tests will be performed at the most aggressive and least aggressive conditions that reasonably can be expected to produce results. This will establish bounds within which the effectiveness of the process can be refined. These tests consist of the following:

1. Process sequence elements:
  - a. Oxidant input sequence:<sup>i</sup>
    - (1) Persulfate only

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i. Current plans are to use Fenton's reagent only. However, if Fenton's reagent is not successful, then this test sequence (which was proposed in a Statement of Work with the Subcontractor) will be used.

- (2) Fenton's reagent only
  - (3) Persulfate/Fenton's/persulfate
  - (4) Fenton's/persulfate
- b. Heating
- c. Catalyst input
- d. Mixing
- 2. Catalyst concentration:
  - a. Persulfate N/A
  - b. Fenton's (Fe:H<sub>2</sub>O<sub>2</sub> wt:wt) 1:1 1:5 1:10
- 3. pH
  - a. Persulfate: unconstrained 2 4
  - b. Fenton's: unconstrained 2 4 6
- 4. Temperature
  - a. Persulfate: 20 80 95
  - b. Fenton's: 20 40 95
- 5. Mixing energy
  - a. Determine a specific mixing rate (watts/liter, shear, flow) at which insufficient mixing begins to diminish reaction progress.
  - b. Conduct all other tests at a higher mixing rate.
- 6. Rate of oxidant addition
  - a. Persulfate: Determine the maximum rate of oxidant addition that the process will tolerate without excessive foaming, heat generation, or the risk of a runaway reaction.
  - b. Fenton's: (same as persulfate).
- 7. Amount of oxidant
  - a. Persulfate: Determine the amount of oxidant in mole ratio of persulfate/total organic compound (TOC) at which the required destruction and removal efficiency (DRE) is met.
  - b. Fenton's: (same as persulfate).
- 8. Surrogate composition
  - a. Average composition of V-Tank contents.
  - b. 95% Upper confidence limit of maximum batch concentration.

## Test Objective 2

To determine kinetic rates for contaminant destruction in the chemical oxidation process. This objective and the following are measurements to be made on both extremes of the process from Test Objective 1 and at suitable points in between. In order to determine kinetic rates, periodic sampling or online monitoring (e.g., pH and temperature) needs to be implemented so that the changes with respect to time can be plotted. This would allow the chemical process to be better understood and provide good estimates for reaction times needed to achieve required extent of reactions.

## Test Objective 3

To estimate the rate of heat generation of the chemical oxidation process.

## Test Objective 4

To determine the gas generation rate of the chemical oxidation process.

## Test Objective 5

To estimate the durability of potential construction materials exposed to the chemical oxidation process. Testing for this objective will involve the use of stainless-steel coupons in the oxidation bath and measuring for pitting and corrosion.

## Test Objective 6

To estimate the changes in leachability between metals in the raw waste and oxidized waste for the chemical oxidation process. Measurement of this change, via the TCLP test, determines potential differences in the leachability of toxic metals due to the oxidation process. Possible changes might occur due to alterations of metallic valence states/speciation. This measurement will provide an indication for the level of metal fixation required for the stabilization step. This information will not be used to re-characterize the waste, but will only be used in comparison with the poststabilization TCLP test to evaluate the effectiveness of the stabilization process. In practice, TCLP analysis for metals would not be mandatory before stabilization. As an experimental measure, useful information might be found.

## Test Objective 7

To determine the sequence and relative rates of organic destruction in the aqueous phase.

## Test Objective 8

To provide data to support a strategy to prevent autocatalytic reactions for both persulfate and Fenton's reagent during active remediation.

## Test Objective 9

To determine the behavior of VOCs in the off-gas, volatilization as opposed to destruction.

Test plan elements, other than the test plan objectives listed above, are outlined in Table 8. Some of these sections will be discussed briefly throughout this work plan. The Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of V-Tank Waste (INEEL 2003a) is more detailed in identifying exact experiments, experimental design, and decision points for further experiments. The flexibility to find a set of less-aggressive test conditions that adequately treat the waste in a suitable reaction time is from the above list of test objectives. If these less-aggressive test conditions are found, further testing at more

aggressive bounding conditions will not be necessary. This type of flexibility allows the laboratory to forgo unnecessary testing.

#### 4.1.2 Test Phase 2

Test Phase 2 testing will consist of a smaller number of tests; it is anticipated that no screening-level tests will be required during Test Phase 2. The parameters for these tests will be determined from the screening tests (Test Phase 1). The purpose of the Phase 2 tests is to use the best condition, as determined from the screening tests, to validate that the chemical oxidation process is successful in reducing the organic contaminants for treatment to target concentrations. The level of rigor associated with quality assurance/quality control is critical in that a successful run will be used to verify that the expected cleanup criteria can be met, in addition to addressing process design requirements. In order to verify that cleanup criteria have been met, stabilization of the postoxidized slurry will need the same rigor type associated with data quality for the metal contaminants to be treated.

Table 8. Test plan elements for the bench-scale cold study.

Test Plan Element	Element Description
Materials and methods	Addresses issues such as grade of reagents, selection of test construction materials, and identification of standard methods for sampling and analysis.
Surrogate composition	Identifies such issues as chemicals and mass of chemicals to be used in the surrogate recipe and the statistical variability of those masses.
Test-scale study	Addresses such issues as the basis for selecting bench-scale test volumes and the scalability of experimentally derived values from the bench scale to field scale.
Method validation testing	Identifies a process for ensuring that the test objectives can be met by ensuring proper mass and energy balances, where necessary.
Identification and sequencing of test parameters	Identifies the parameters to be optimized and discusses the methodology for selecting these test parameters.
Statistical-based test design	Identifies the basis for and content of a formalized test plan design, including the number of tests required, the number of samples to be acquired, and methods to be used to statistically treat collected data to ensure that the objectives of the Cold Bench-Scale Test Plan (INEEL 2003a) are met.
Safety and health	Identifies necessary elements of the required safety and health guidelines established at MSE Technology Applications.
Environmental/residuals management	Includes a plan for disposal of test-related materials.
Quality assurance/quality control	Identifies the quality levels of each type of data collected and ensures that proper plans are in place to produce defensible and reliable data to support V-Tank remediation. Includes methods for handling, analyzing, and interpreting data.
Traceability matrix	Includes the means to track from every data point collected to the test plan objective driving the collection of those data.
Schedule	Includes dates for milestones and completion of the test project.

## **4.2 Test Phase 3—Experimental Design and Procedures for Bench-Scale Hot Study**

The purpose of Test Phase 3 (the bench-scale hot tests) is to reproduce the results from Test Phase 2 on an actual V-Tank contents' sample to validate success in achieving the expected cleanup criteria. This work will commence shortly after Test Phase 2 (tests on surrogate samples) is completed. A high level of data assurance/quality control is required for Test Phase 3.

As part of Test Phase 3, the subcontracted laboratory will repeat surrogate testing using the same optimum test conditions as used in Test Phase 2. This replicate surrogate test will baseline the process and will serve as an independent verification. If results from runs with the actual V-Tank waste vary sharply from the cold test results, this should be the result of differences between the nature of the actual tank waste and the surrogate used to mock the waste, and not between the two subcontracted laboratories used to perform the tests. This hot test will be performed with samples gathered in FY 2004 as part of a tank-content consolidation effort that is in the planning stages. If consolidation of tank contents cannot occur in FY 2004, previously sampled V-Tank contents that are in storage at TAN, as investigation-derived samples, will be utilized for the hot tests. There is expected to be depleted amounts of TCE, tetrachloroethene (PCE), and TCA in these samples, which will require the subcontracted laboratory to spike these samples to levels similar to levels that will be found in the actual mixed V-Tank waste. Guidance from BBWI will provide the laboratory with instructions on how to proceed with any sample modification.

The subcontractor performing the experiments for Test Phase 3 (bench-scale hot studies) will prepare the test plan. This subcontractor will need to coordinate with MSE Technology Applications and BBWI to write these procedures. Test Phase 3 is slated for FY 2004.

## **4.3 Test Phase 4—Experimental Design for the Full-Scale Tests**

Test Phase 4, the full-scale cold mockup study, will commence after Test Phases 2 and 3 are completed. The purpose of the full-scale cold mockup tests is to use the optimum conditions of these previous test phases to investigate issues related to scale-up and to ultimately prepare a unit for an SO test. Temperature rises, sampling constraints, and off-gas-system component integration can be investigated at larger scale, in addition to determining whether necessary organic destruction efficiencies have been achieved. Before the final SO tests, the number of tests planned for Test Phase 4 also will depend on the results of both Test Phases 1 and 2.

It is anticipated that MSE Technology Applications will prepare the Test Phase 4 test plan. Test Phase 2 (the bench-scale cold study) and the BBWI remedial design/remedial action work plan (finalized in the middle of FY 2004) will supply information needed for the Test Phase 4 test plan. Test Phase 4 will begin in FY 2005.

## **5. TEST LOCATION, EQUIPMENT, AND MATERIALS**

### **5.1 Bench-Scale Cold Tests (Test Phases 1 and 2)**

#### **5.1.1 Test Location of Bench-Scale Cold Tests**

The bench-scale cold tests will be performed under a subcontract at facilities operated by MSE Technology Applications in Butte, Montana. The tests at MSE Technology Applications will be performed with surrogates. These facilities are currently operational, and they will have the necessary equipment to conduct the proposed experiments. Instrumentation, laboratory hoods, and other related equipment are provided to ensure safe completion of experiments.

The test plan document will be co-authored by MSE Technology Applications and BBWI; this document will describe in detail the test equipment and materials that MSE Technology Applications will use.

#### **5.1.2 Bench-Scale Cold Test Equipment**

The primary components used for bench-scale cold tests include the following:

- Reactor vessel
- Stirrer
- Peristaltic pumps
- Cement mixer.

An extensive component list will be provided in the upcoming test plan.

#### **5.1.3 Bench-Scale Cold Test Materials**

The primary equipment used for bench-scale cold tests include the following:

- Simulated waste (surrogate)
- Oxidants (i.e., sodium persulfate, hydrogen peroxide, and ferrous salt)
- Caustic (i.e., sodium hydroxide and lime)
- Grout additives (i.e., Portland cement, fly ash, and blast furnace slag).

### **5.2 Bench-Scale Hot Tests (Test Phase 3)**

#### **5.2.1 Location of Bench-Scale Hot Test**

The bench-scale hot tests will be performed off-Site at a suitable laboratory. One such candidate laboratory is the Lynchburg Research Center (LRC) facilities operated by BWXT Inc., in Lynchburg, Virginia. A previous treatability study on V-Tank waste using chemical oxidation and stabilization was conducted at their facilities (INEEL 1998). These tests will be performed with samples of actual V-Tank material. The facilities at LRC are currently operational, and they will have the necessary equipment to conduct the proposed experiments. In addition, these facilities have the necessary permits and safeguards

to work with radioactive hot samples. Instrumentation, laboratory hoods, and other related equipment are provided to ensure safe conduct of experiments.

### **5.2.2 Bench-Scale Hot Test Equipment**

The test equipment used in the bench-scale hot test study should be identical to the equipment used in the bench-scale cold test study.<sup>j</sup> Since the results of the cold test study feed into this study, the equipment list in this report is considered as to-be-determined (TBD) status. A test plan, co-authored by LRC and BBWI, will detail the necessary laboratory equipment and materials.

### **5.2.3 Bench-Scale Hot Test Materials**

The following materials will be used in the bench-scale hot test:

- Simulated waste (surrogate)
- Actual V-Tank sample (from Tanks V-1, V-2, V-3, and V-9)
- Oxidants
- Caustic
- Grout additives (i.e., Portland cement, fly ash, and blast furnace slag).

## **5.3 Full-Scale Cold Mockup Tests (Test Phase 4)**

### **5.3.1 Location of Full-Scale Test**

The full-scale cold mockup tests will be performed off-Site at a suitable location. One candidate facility under consideration is MSE Technology Applications in Butte, Montana. These tests will be performed at the actual treatment scale so that effects of process-scale (i.e., mixing and heat of reaction) can be determined. The facilities at MSE are currently operational, and they will have the necessary high bay space to conduct the proposed experiments. Instrumentation, laboratory hoods, and other related equipment are provided to ensure safe conduct of experiments. The design/drawings of the full-scale unit will be finalized in the upcoming remedial design/remedial action work plan.

### **5.3.2 Full-Scale Test Equipment**

The test equipment used in the full-scale cold mockup test will hinge on the results of the cold test study and the remedial design/remedial action work plan. A test plan, co-authored by MSE Technology Applications and BBWI, will detail the experimental design and the necessary components of the test equipment and test materials. The following is a brief list of some of the components:

- Full-scale reactor vessel
- Mixer

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j. Some equipment might need to be different or altered, depending on the configuration required for radioactive handling for dose or contamination concerns.

- Pumps for liquids and slurries
- Full-scale grout unit.

### **5.3.3 Test Materials**

The following is a list of test materials:

- Surrogate tailored for full investigation
- Oxidant
- Caustic solution
- Grout additives.

## 6. SAMPLING AND ANALYSIS

A Sampling and Analysis Plan (SAP) will be issued as part of Test Phases 1 and 2. The SAP will describe the sampling and analysis procedures and specific data quality objectives/levels for this preliminary work plan study. The SAP will be used to ensure that proper sampling and analysis procedures are used to provide valid and reliable test results.

A tank-contents consolidation effort currently is being planned for FY 2004. Samples of V-Tank contents would be taken during this effort and utilized for bench-scale hot tests. The sampling plan for obtaining these samples would be part of the documentation for that effort. In the event that the consolidation activity does not occur in FY 2004, unused samples of the tank contents currently in storage at TAN would have to be used. There is assumed to be a sufficient supply of unused tank contents that exist as investigation-derived waste, currently inventoried at TAN-607, that can be used in this study. Table 9 shows the current inventory of unused samples.

Table 9. Current inventory of V-Tank samples that can be used for Test Phase 3.

Tank Identification	Liquid Quantity (L)	Sludge Quantity (L)	Liquid/Sludge <sup>a</sup> (L)
V-1	8.3	9.1	2.0
V-2	0	11.5	1.3
V-3	0	0.7	2.3
V-9	0	1 to 8	0
Combined <sup>b</sup>	N/A	N/A	29.5

a. The supernatant liquid and sludge are combined within the same sample bottle.

b. Combined refers to a composite sample from Tanks V-1, V-2, and V-3.

As shown in Table 9, enough unused tank contents exist to perform experiments. The containers of unused tank contents will be pooled together and sent to BWXT in Lynchburg, Virginia, to conduct the hot-phase tests of the preliminary design and laboratory work plan study. A SAP, or other applicable documentation, will be put together to address the consolidation and shipment of samples to BWXT. The purpose of consolidation is to provide the best representation of what is anticipated to be the worst-case feed to the treatment process. Previous attempts at chemical oxidation and stabilization of V-Tank waste are described in *Final Report Treatability Study for LMITCO TSF-09 V-1, V-2, and V-3 Tank Waste* (INEEL 1998).

## 7. DATA MANAGEMENT

Data generated during testing will be managed in accordance with guidelines provided in the *Data Management Plan for the Idaho National Engineering Laboratory Environmental Restoration Program* (INEL 1995a). This plan provides or references procedures and requirements necessary to develop a database of relevant information, which can be readily accessible and accurately maintained. The Data Management Plan describes the data flow process, data custodianship, and organizational and individual responsibilities associated with data management. The Data Management Plan also provides the project file and reporting requirements, and it identifies extensive database-capability requirements to allow selective data sorting, analysis, formatting, and reporting. In addition, hard copies of the raw data and test results will be summarized in the final preliminary work plan study report.

The Data Management Plan (INEL 1995a) provides the necessary requirements for this preliminary work plan study. The Data Management Plan is currently up for revision, and there may be some deviations from the plan. Deviations are caused by the experimental nature of the work, wherein some information might not be directly recorded in logbooks or from some laboratory data that are not tracked by the Environmental Data Warehouse.

Hard copies of raw data and test results will be summarized in Engineering Design Files (EDFs) following each scheduled test. Data quality objectives and data validation requirements are provided in this Laboratory Study Work Plan and the Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of V-Tank Waste (INEEL 2003a).

The management control procedures (MCPs), plan (PLN), and program requirements document (PRD) that will be used to manage the data generated from this work plan study include the following:

- MCP-233, "Process for Developing, Releasing, and Distributing ER Documents (Supplemental to MCP-135 & MCP-9395)"
- MCP-238, "Test Plans"
- MCP-240, "ER/D&D&D Operational Review Board Process"
- MCP-1194, "Logbook Practices for ER and D&D&D Projects"
- MCP-3480, "Environmental Instructions for Facilities, Processes, Materials, and Equipment"
- PLN-476, "Records Management Plan for the Environmental Restoration Program and Projects"
- PRD-5030, "Environmental Requirements for Facilities, Processes, Materials, and Equipment."

## **8. DATA ANALYSIS AND INTERPRETATION**

After completion of the preliminary design and laboratory study work plan's experimental phase, the data will be summarized, analyzed, and interpreted to determine the validity and performance of the treatment process. One goal will be to determine the quality of the data collected. Designated analytical data will be checked to assess the precision, accuracy, and completeness. If the quality objectives are not met, consensus on a path forward will be sought with BBWI (management and operations contractor), the U.S. Department of Energy Idaho Operations Office (DOE-ID); the U.S. Environmental Protection Agency (EPA), Region 10; and the Idaho Department of Environmental Quality.

The data will be both qualitative and quantitative. Qualitative data will include photographic records of the equipment set-up and surrogate appearance, logbook entries, descriptions, and sketches. Quantitative data will include measurements of the amounts of surrogate and oxidant used, chemical concentration measurements, laboratory analysis, and results. Data of sufficient quality will be obtained from the work plan study to evaluate the effectiveness and implementability for possible full-scale testing or actual treatment of V-Tank waste.

Test results will be interpreted in the context of the technology's effectiveness, implementability, and costs. Project personnel will summarize the raw data and test results in EDFs at the end of the laboratory tests. The EDFs will provide the key information needed for complete data analysis and interpretation in this preliminary design and laboratory study.

### **8.1 Data Quality Levels**

Two levels of data quality will be used in this study. Data taken for preliminary selection or adjustment of mix parameters (and not to be relied upon in design analysis) will be taken in the screening mode. This corresponds to a category of data known as "Screening Data with Definite Confirmation" in the EPA classification system. In this work plan, data gathered in Test Phase 1 fall into this category. In order to permit a broad range of data to be gathered at reasonable cost, screening data will be collected from typical single specimens, and single-value measurements will be made in accordance with detailed written procedures. Results will be presented on standard report forms used by the specific contractor. Comparisons with values established as test target goals will be made on a pass/fail basis using estimated allowances for data uncertainty bands. This mode will be applied to all tests that deal with preliminary selection or adjustment of parameters and to peripheral data taken in later phases. These data are not intended for use as a basis for design evaluations or future full-scale design applications.

A second level of data quality will be used for data that are gathered directly for design analysis purposes or as a critical element of a full-scale treatment process. Data taken at this quality level will be developed using definitive test methods. This corresponds to a category known as "Definitive Data" in the EPA classification system. All specimen preparation activities will be conducted in accordance with detailed written procedures, and all measurements included in the final report will be taken with instruments calibrated to certified standards. This second level of data quality will be exercised for all testing in this work plan except for Test Phase 1.

Complete data packages from each phase of the preliminary design and laboratory study will be sent to BBWI upon completion of the laboratory work associated with that phase. In addition to the analytical data collected during the study, data packages also will contain relevant observations of key parameters and unknowns encountered during testing. The subcontractor will perform all tests in accordance with the appropriate test plans and approved laboratory procedures. The data resulting from testing and analysis under each phase will be collected and reported to BBWI in their entirety. The subcontractors, in addition to the subject-matter experts, will offer technical interpretation of specific test

results. The final decision on selection of the best set of conditions for treatment based on the overall data will be made by BBWI.

## **8.2 Quality Assurance**

The quality of the activity is highly dependent on the technical expertise of the performers and technical oversight by BBWI project personnel. For this reason, BBWI technical representative should review the résumés of subcontracted personnel performing this work to ensure that appropriate personnel are assigned to the work and any technical guidance is provided from BBWI as the research activity progresses.

For Test Phase 1 and 2, since line implementation of the MSE Quality Assurance Program is implemented by an activity-specific MSE quality plan, review of the MSE quality plan for this activity should be provided to BBWI for review and comment as well as concurrence to the MSE assigned data-quality level.

## **9. HEALTH AND SAFETY**

### **9.1 Bench-Scale Cold Study**

The subcontractor should have their own health and safety plan for work to be performed at their laboratories. Cognizant safety personnel from BBWI can assist in preparing or reviewing this plan to ensure that the execution of the Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of V-Tank Waste (INEEL 2003a) can be performed safely.

Health and safety issues center around the following:

- Preparation of the surrogate, involving the addition of quantities of solvent (i.e., TCE, PCE, and TCA), mercury, and chemical reagents (i.e., PCB and bis [2-ethylhexyl] phthalate)
- Uncontrolled oxidation resulting from conditions that promote rapid reaction rates.

Other health and safety issues involve uncontrolled off-gas emissions, static electricity buildup on system components, rapid heat buildup, hazards of heating peroxides, over-pressurization of reaction vessel and ventilation system, and saturation of off-gas blowing through ventilation. The health and safety plan prepared in conjunction with the Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of V-Tank Waste (INEEL 2003a) should outline the hazards and mitigations for these hazards.

### **9.2 Bench-Scale Hot Study**

The laboratory safety and health concerns for the bench-scale hot study will be addressed by BWXT Services, Inc., in Lynchburg, Virginia. In 1998, the LRC performed chemical oxidation of V-Tank samples for the INEEL (INEEL 1998). They were covered by a health and safety plan at that time to perform the aforementioned work. If chosen for this study, the INEEL will ensure that their health and safety plan is still valid for this work and complies with the Occupational Safety and Health Administration standards, including 29 CFR 1910.120, "Hazardous Waste Operations and Emergency Response," and 29 CFR 1910.1450, "Occupational Exposure to Hazardous Chemicals in Laboratories." By applying the necessary safety and health procedures, the various chemical and physical hazards associated with this specific preliminary design and Laboratory Study Work Plan will be minimized. Each area supervisor and manager involved in this preliminary design and Laboratory Study Work Plan maintains a copy of the manual, and their personnel are trained in applicable procedures.

The specific requirements for the subcontractor performing chemical oxidation for the INEEL will be detailed in a future Statement of Work (SOW). All radiological issues associated with the Laboratory Design Study Work Plan shall be covered in LRC's various area operating procedures. Any work not covered by an existing procedure shall be performed under a radiological work permit. Personnel must be trained on the specific radiological work permit under which they are working. All LRC personnel receive annual site-specific radiation worker training from trained facility health physicists. The LRC is listed in the INEEL's Qualified Supplier List; the LRC has worked with radiologically hot samples from the INEEL.

### **9.3 Cold Full-Scale Study**

The subcontractor should have their own health and safety plan for the work to be performed at their facilities. Data from earlier test phases will be used to ensure that the full-scale study can be executed safely.

## 10. MANAGEMENT OF RESIDUALS

Because MSE will use surrogate waste forms, there will be no involvement of actual V-Tank samples from either the bench-scale cold test or the full-scale test. All waste management activities will be the responsibility of MSE. In addition, BBWI must ensure that the subcontractor has a waste management plan in place for disposition of laboratory waste and residuals before conducting experiments that are detailed in this Laboratory Study Work Plan and the Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of V-Tank Waste (INEEL 2003a).

The following residual waste might be generated from the bench-scale hot test during testing:

- Unused, untreated V-Tank waste samples
- Unused, spiked V-Tank waste samples
- Pretreated waste
- Solidified waste forms
- Treatment residuals
- Extraction fluids
- Used sample containers
- Contaminated protective clothing and other personal protective equipment
- Decontamination fluids.

Currently, the altered and unaltered V-Tank samples from previous sampling events are residing at TAN-607. These samples will be recombined into samples that will be representative of the worst-case feed that the ES-CO/S process would incur. The recombination will take place either at the INEEL or at LRC. If performed at LRC, recombination guidance will be provided. In either case, waste (such as personal protective equipment, wipes, smears, and containers [glass and plastic]) will be generated.

If consolidation of the V-Tank waste samples is performed at the INEEL, hazardous, mixed low-level waste, low-level radioactive waste, and industrial waste will be generated. In addition to the F001 waste code, these types of waste might contain contaminants (such as PCBs) that could be regulated by the Toxic Substances Control Act (15 USC § 2601 et seq.). These types of waste may be disposed of at the INEEL if they meet facility-specific waste acceptance criteria. Typically, most of these CERCLA-generated waste types will be sent to the ICDF for disposal. This waste is required to meet the ICDF's waste acceptance criteria, which are delineated in the following report: *ICDF Complex Waste Acceptance Criteria* (DOE-ID 2002a). In addition, both hazardous and mixed low-level waste must meet applicable RCRA land disposal restrictions. However, CERCLA-generated industrial waste typically is disposed of at the INEEL Landfill Complex. The use of the Radioactive Waste Management Complex is an option for disposal of suitable CERCLA-generated low-level radioactive waste.

Some of the waste generated during CERCLA remedial activities may be sent to a treatment, storage, and disposal facility outside of the INEEL boundaries. The CERCLA hazardous or mixed waste that is sent off-Site for treatment, storage, or disposal may only be sent to a permitted or interim status treatment, storage, and disposal facility that is suitable to receive hazardous waste from CERCLA remediation sites by the treatment, storage, and disposal facility's own EPA regional office in accordance with 40 CFR 300.440(a)(4).

At the subcontracted facility (most likely the LRC), tank waste materials will be characterized, chemically oxidized, and stabilized during the study. Countertops will be covered with blotter paper or plastic before preparing the materials for oxidation, stabilization, and analytical testing. Disposable gloves will be worn during waste and specimen handling, and rags and wipes will be used for decontamination. These materials could become contaminated waste upon contact with tank material; thus, they will be combined into a mixed low-level waste (MLLW) debris waste stream. Items, such as glassware and pipettes that cannot be decontaminated, also will be included in a MLLW debris waste stream. Table 10 shows the estimated quantities of solid waste produced during field and laboratory activities.

Industrial and contaminated waste also will be generated at the preliminary design and laboratory study location. The subcontractor will be responsible for segregating industrial waste from contaminated waste and for managing the industrial waste. Several types of contaminated waste also might be generated during these operations. Segregation of the contaminated waste will be required to facilitate subsequent waste management. Any remaining unaltered tank samples will be segregated from all other waste. Likewise, any laboratory study product that has not been altered by testing will be segregated from all other waste. All other waste contaminated by contact with V-Tank contents must be segregated by debris/solidified waste forms and waste containing free liquid. Any waste that is generated from contact with contaminants other than V-Tank contents will be segregated. The subcontract laboratory must segregate this waste and develop their own segregation protocols, which are required to meet the protocols established in an upcoming SOW, the future Test Phase 3 Study Test Plan, and the waste categories identified in the *Idaho National Engineering and Environmental Laboratory Waste Acceptance Criteria* (DOE-ID 2003b).

A portion of the V-Tank waste sent to the laboratory for laboratory study will be used in analytical testing, and the rest of the V-Tank waste will be treated during different phases of testing. A portion of the treated V-Tank waste will be analyzed; the remainder will be considered waste. The quantity of material used for analysis during initial characterization and following treatment will vary depending on the actual tests, dilutions, and reruns performed. Analytical testing will affect the physical and chemical properties of prepared samples. The sample volume could increase or decrease based on the analysis. During the preliminary design and laboratory study, equipment that will be reused in the laboratory must be decontaminated. An aqueous solution will be used for general radioactive decontamination purposes. Table 10 shows the estimated quantities and types of waste that will be produced during the treatments, analytical testing, and decontamination activities at the laboratory. When the subcontracted laboratory is identified and a test plan for the laboratory activities is prepared, this test plan shall contain an updated waste generation table that will supercede Table 10.

All waste identified in Table 10 is considered to be solid waste in accordance with RCRA criteria cited in 40 CFR 261.2, "Definition of Solid Waste." The "Resource Conservation and Recovery Act (Solid Waste Disposal Act)" (42 USC § 6901 et seq.) defines a solid waste as a solid, liquid, or contained gas discarded by being abandoned or recycled; or that is inherently waste-like. In addition, RCRA requires that a hazardous waste determination be performed on all solid waste.

At a minimum, the V-Tanks' contents are regulated under RCRA for F-listed spent solvents (F001—trichloroethylene). Therefore, all solid waste generated during the bench-scale hot study that has contacted the V-Tank contents becomes a mixture of solid waste and a hazardous waste, and it will inherit the F-list code.

Table 10. Test Phase 3 waste generation estimates.<sup>a</sup>

Activity Description	Waste Description	Expected Type	Estimated Quantity	Planned Treatment/Disposal
Sample consolidation	Secondary waste, personal protective equipment, wipes, smears, and compositing equipment	MLLW (F001)	70 gal	Macro or microencapsulate/ICDF <sup>b</sup>
Preliminary design and laboratory study	Aqueous, acidic from decontamination activities	MLLW (F001, D002)	75 L	Neutralization/stabilization/ICDF <sup>b</sup>
Preliminary design and laboratory study	Aqueous, basic or neutral from decontamination activities	MLLW (F001)	70 L	Stabilization/ICDF <sup>b</sup>
Preliminary design and laboratory study	Solvent extraction residuals (acetone, methylene chloride, and methanol) from organic analyses	MLLW (F001 and other designate F-codes)	2 L	Chemical oxidation with the V-Tank contents/stabilization/ICDF <sup>b</sup> or treatment at off-Site commercial vendor
Preliminary design and laboratory study	Solid, sample residues, basic or neutral	MLLW (F001)	15 L	Stabilization/ICDF <sup>b</sup>
Preliminary design and laboratory study	Solid, sample residues, acidic	MLLW (F001, D002)	0.2 L	Neutralization/stabilization/ICDF <sup>b</sup>
Preliminary design and laboratory study	Solid, debris	MLLW (F001)	30 gal	Macro or microencapsulate/ICDF <sup>b</sup>
Preliminary design and laboratory study	Solid, excess concrete sample	MLLW (F001)	12 L	Expected to meet treatment standards/ICDF <sup>b</sup>

a. The U.S. Department of Transportation packaging is expected to be within U.S. Department of Transportation-compliant containers. The expected storage location will be a CERCLA waste storage area.

b. The ICDF is the planned disposal facility. However, other facilities such as the Radioactive Waste Management Complex or Envirocare, may be used for disposal (as appropriate).

CERCLA = Comprehensive Environmental Response, Compensation, and Liability Act

ICDF = INEEL CERCLA Disposal Facility

MLLW = mixed low-level waste

## **10.1 Waste Minimization and Segregation**

Waste minimization will be achieved through design and planning to ensure efficient operations and that waste is not generated unnecessarily. The earlier bench-scale cold study will identify the specific experiments that need to be run to verify the proper oxidation recipe and stabilization techniques to achieve a disposable waste form. This will naturally limit the amount of hot testing required for the subcontractor.

## **10.2 Waste Management and Disposition**

The subcontracted laboratory will be responsible for managing contaminated waste generated at the laboratory while conducting the Test Phase 3 study, before returning the contaminated waste to the INEEL. To initiate the return of this waste to the INEEL, the subcontracted laboratory will notify BBWI by a written report identifying the volumes and characteristics of each waste type, including shipping and packaging details. Most of the information required for this report will be provided by completing Form 435.28, "Solid/Hazardous Waste Determination"; BBWI will assist the subcontractor in preparing these forms. Final authorization for the return of waste will be provided to the subcontractor in writing from BBWI. Contaminated waste will be sent to a CERCLA storage area at the INEEL for storage. Unaltered samples could be returned immediately to the V-Tanks as investigation-derived waste, because the returned material will be treated with the source material in the tanks during final remediation. The SOW and test plan will be in place at the beginning of the next fiscal year. All waste management activities undertaken by the subcontract laboratory must be performed in accordance with this plan, the SOW, and the test plan.

Further information that discusses disposal strategies for CERCLA waste from WAG 1 activity is provided in the *Waste Management Plan for V-Tanks Early Remedial Action for the Test Area North, Waste Area Group 1, Operable Unit 1-10, Group 2 Sites* (INEEL 2003b). This document discusses the various disposal options on-Site and addresses the appropriateness of management and disposal option for anticipated waste.

## 11. COMMUNITY RELATIONS

This community relations task is designed to ensure community understanding of actions taken during the preliminary design and laboratory study and to obtain community input on the preliminary design and laboratory study program. Community relations are an integral part of any CERCLA action, regardless of whether the action is at a federal facility. All CERCLA actions at the INEEL will be subject to CERCLA community involvement requirements. The INEEL Public Affairs group prepared the programmatic *Community Relations Plan—A Guide to Public Involvement in the Environmental Restoration Program at the INEL* (INEL 1995b) that covers the INEEL's remedial investigation/feasibility study process. The Community Relations Plan was issued as a U.S. Department of Energy (DOE) document representing "the process established by mutual agreement between the DOE, EPA, and the State of Idaho to address ER concerns at the INEEL." The plan will be used as guidance for any actions taken to ensure appropriate public involvement. If necessary, WAG 1 will support news and press releases and will actively relay test plans and preliminary design and laboratory study results at public meetings.

## 12. REPORTS

The following report practices should be followed in all test phases discussed in this work plan. The principal investigator, with assistance from the lead engineer, is responsible for submitting monthly reports that describe the progress of the preliminary design and Laboratory Study Work Plan. As a minimum, the monthly reports will be submitted to:

- The project manager (responsible for the V-Tanks)
- The project engineer (responsible for the V-Tank preliminary design and laboratory study)
- The project engineer (responsible for the V-Tank Conceptual Design Report)
- The process engineer (responsible for the chemical process of the V-Tank conceptual design)
- The TAN system engineer
- The WAG 1 e-mail file (under the guidance of Document Control).

Monthly reports should include, but not be limited to, the following:

- Accomplishments of the work performed for the month
- Anticipated work to be performed for the following month
- Any problems or issues encountered and the actions taken.

Interim support documents for this effort include a test plan that may contain a SAP, a health and safety plan or facility equivalent, and a waste management/minimization plan. These contractor facility-specific inclusions to the test plan should be available for review by BBWI, if not specifically added to the test plan.

The subcontractor performing the preliminary design and laboratory study will document study activities by preparing the following reports:

- Brief letter or e-mail status reports documenting the preliminary performance of specific treatment technologies.
- An interim report documenting studies, procedures, and findings that will be issued at the completion of key findings, as determined by BBWI. This report may eventually be incorporated into the final report. The interim report may be used as a basis for DOE-ID/BBWI to authorize additional design-study work-plan activities.

Sections of the test plan will describe additional interim reporting requirements. At the completion of specific design and laboratory-study work-plan activities, the subcontractor performing the work will prepare a report documenting project activities and findings.

After completion of work, the INEEL will review the interim and final reports. The subcontractor will be available to address comments generated during the reviews. Table 1 shows an example of the suggested Design Study Work Plan report format that may be used as a basis for preparing the interim or final Preliminary Design Laboratory Study Work Plan.

Table 11. Suggested final report format.<sup>a</sup>

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1.	INTRODUCTION
1.1	Site Description
1.1.1	Site Name and Location
1.1.2	History of Operations
1.1.3	Past Removal and Remediation Activities
1.2	Waste Stream Descriptions
1.2.1	Waste Matrices
1.2.2	Pollutants/Chemicals
1.3	Treatment Technology Description
1.3.1	Treatment Process and Scale
1.3.2	Operating Features
1.4	Previous Preliminary Design Study Work Plans at the Site
2.	CONCLUSIONS AND RECOMMENDATIONS
2.1	Conclusions
2.2	Recommendations
3.	PRELIMINARY DESIGN AND LABORATORY STUDY WORK PLAN APPROACH
3.1	Test Objectives
3.2	Experimental Design and Procedures
3.3	Equipment and Materials
3.4	Sampling and Analysis
3.4.1	Waste Streams
3.4.2	Treatment Process
3.5	Data Management
3.6	Deviations from the Work Plan
4.	RESULTS AND DISCUSSIONS
4.1	Data Analysis and Interpretations
4.1.1	Analysis of Waste Stream Characteristics
4.1.2	Analysis of Preliminary Design and Laboratory Study Data
4.1.3	Comparison to Test Objectives
4.2	Quality Assurance/Quality Control
4.3	Costs/Schedule for Performing the Preliminary Design and Laboratory Study Work Plan
4.4	Key Contacts
5.	REFERENCES
6.	APPENDIXES
A.	Data Summaries
B.	Standard Operating Procedures

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a. Reference: EPA 1992, *Guidance for Conducting Treatability Studies under CERCLA*.

Upon submittal of the final report, a complete data package from the design work plan will be kept for record storage. This shall include a copy of all logbook entries during the tests, laboratory monitoring records, files, operational data files, and sampling and analysis files.

### 13. SCHEDULE

This section presents the proposed schedule for the V-Tank preliminary design and laboratory study work plan (see Figure 2). The working schedule shows the relationship of the preliminary design and laboratory study work plan. Figure 2 provides a general schedule for all of the test phases.

Test Phase 3 will commence at the middle of FY 2004, pending initiation of the tank-contents consolidation effort. If existing unused samples at TAN are used instead, Test Phase 3 could begin much sooner (early FY 2004). The final report for this test phase should be complete by the end of FY 2004. The LRC is scheduled to perform the work for Test Phase 3.

Test Phase 4 also will commence by early FY 2005. The final report for this test phase should be complete by the end of FY 2005. The MSE Technology Associates is scheduled to perform the work for Test Phase 4.

Table 12 identifies the deliverables and working schedule dates for this work plan and the study reports that will be submitted to the Agencies for review or information.

Table 12. Deliverables and working schedule dates for this work plan and the study reports that will be submitted for Agency review.

Deliverable	Planned Submittal Date	Review Duration (calendar days)	Document Type <sup>a</sup>
Draft Laboratory Study Work Plan <sup>b</sup>	May 15, 2003	30	Secondary
Cold Laboratory Test Report	TBD	30	Secondary
Hot Laboratory Test Report	TBD	30	Secondary
Cold Mockup Test Report	TBD	30	Secondary

a. The document types (primary and secondary) are described in detail in the Technology Evaluation Scope of Work for the V-Tanks, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10 (DOE-ID 2002b).

b. Previously identified in the Technology Evaluation Report (DOE-ID 2003a) as the Preliminary Design Study Work Plan. Title is changed to be more descriptive.

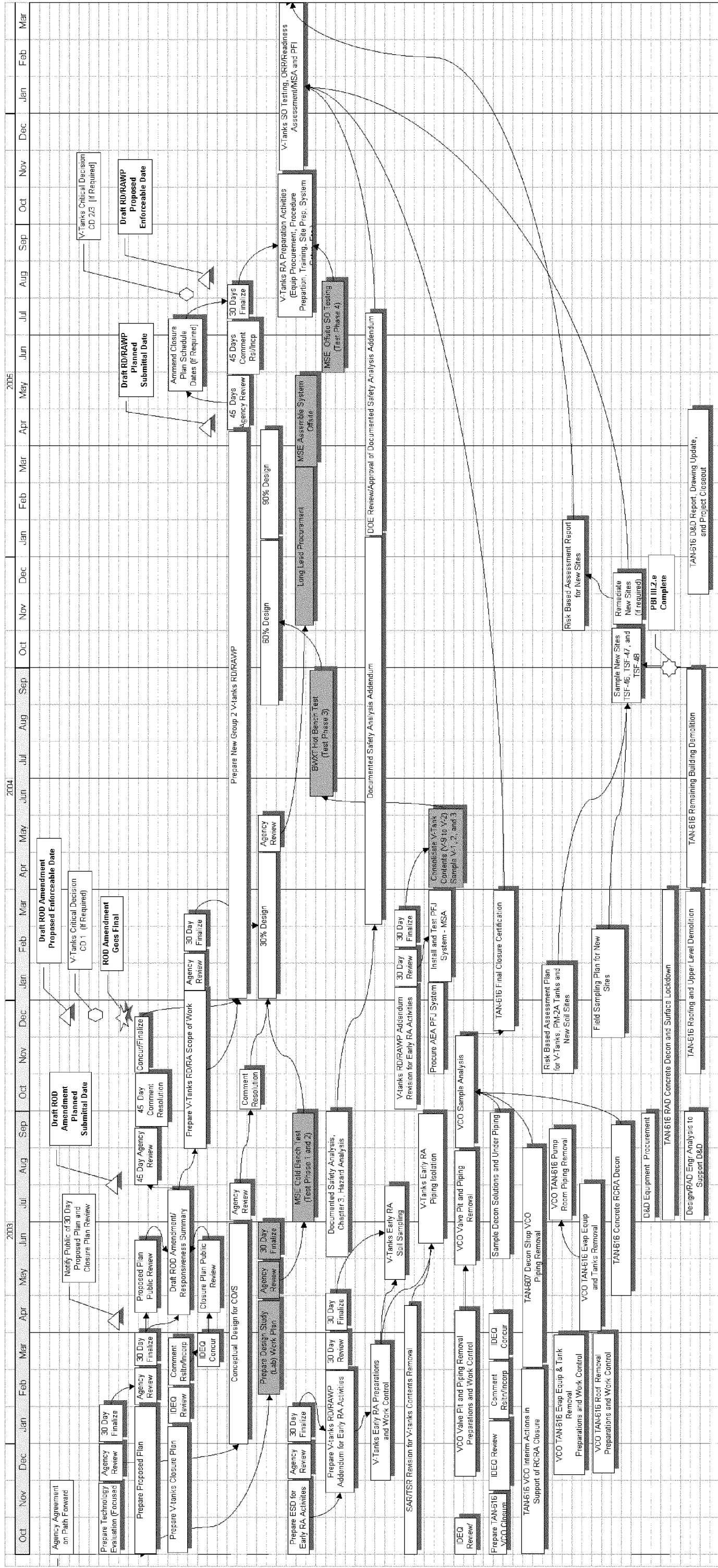


Figure 2. Schedule for Test Phases 1 and 2.

## **14. MANAGEMENT AND STAFFING**

The V-Tank preliminary design and laboratory study work plan's organizational structure reflects the resources and expertise required for performing the work and producing a technically superior product, while minimizing risks to worker health and safety. Figure 3 is an organizational chart for the planned work. A subcontracted laboratory, MSE Technology Applications, will be directly responsible for performing Test Phases 1, 2, and 4 and reporting the results to BBWI. Bechtel BWXT Idaho, LLC, has the ultimate responsibility for the technical quality of the work performed. Bechtel BWXT Idaho, LLC, personnel are responsible for providing the information regarding the surrogates that best capture the state of the V-Tank contents.

### **14.1 Bechtel BWXT Idaho, LLC, Management and Staffing**

The BBWI management-control system uses a three-tier management chain at the contractor level, as directed by Agency, WAG, and program managers. The principals involved in this study include the WAG program manager, the project manager, the project engineer, the principal investigator, and the lead engineer. Other roles that relate to the preliminary design and laboratory studies are the quality assurance engineer, the regulatory compliance engineer, the health and safety engineer, Document Control coordinator, and the principal TAN engineer. The project engineer and the process engineer from the V-Tanks preliminary design and laboratory study will have an interface with respect to the study, as they will supply input (such as data quality objectives).

### **14.2 Subcontractor Management and Staffing for the Laboratory Study Work Plan**

The laboratory studies will be performed at MSE Technology Applications, which is a DOE facility in Butte, Montana. The MSE Technology Applications personnel, under the directions of an MSE Technology Applications project manager, will perform the work for Test Phases 1, 2, and 4. A simple, hierarchical organization structure for this project is shown in Figure 3.

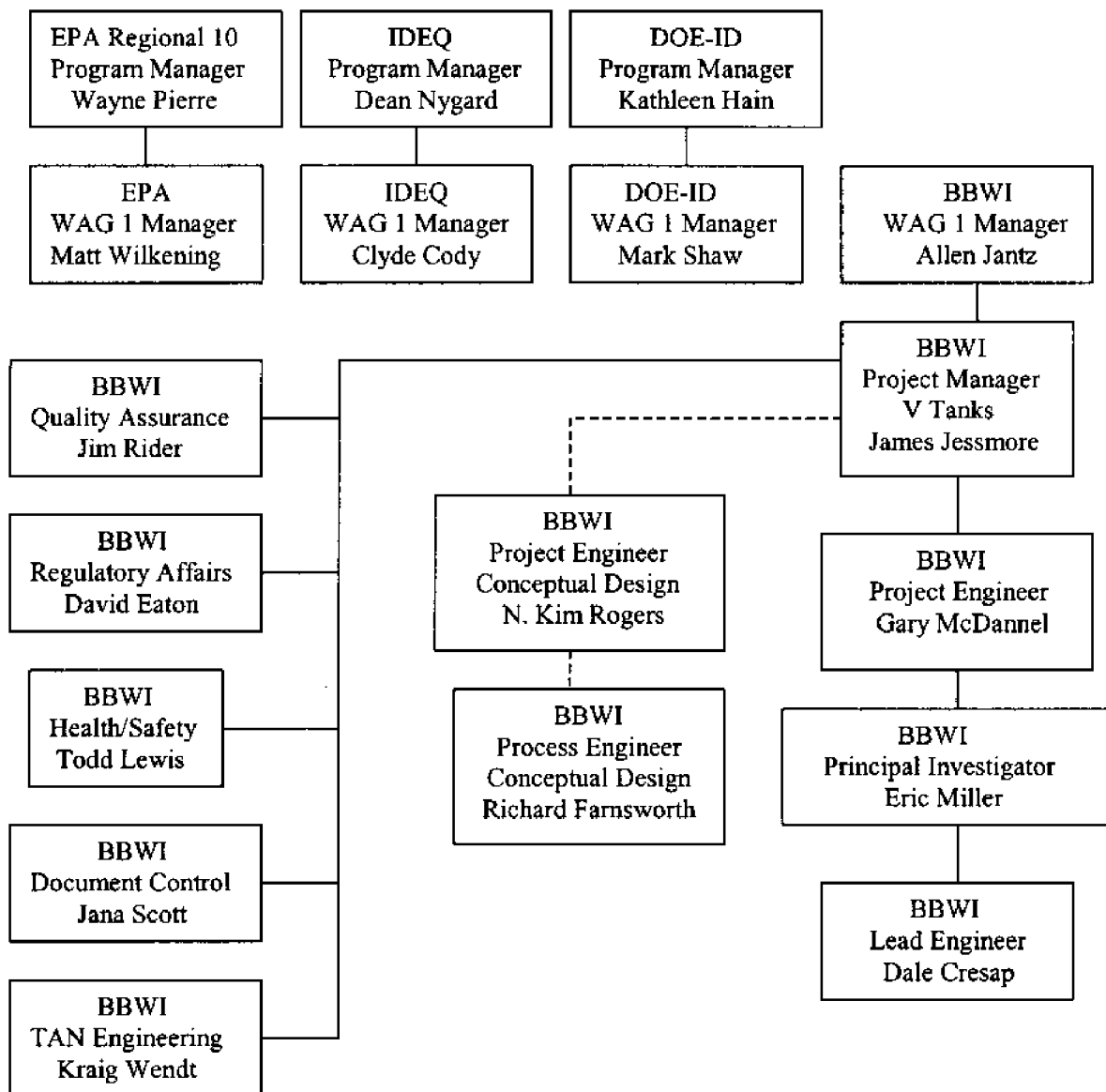


Figure 3. Organizational chart for the chemical oxidation/stabilization preliminary design and laboratory study work plan for V-Tank waste.

## 15. BUDGET

The budget for this work plan is divided among the four phases. Test Phases 1 and 2, the bench-scale cold study, will be funded through both WAG 1 and MSE Technology Applications through an Intragovernmental Payment and Collection System agreement. This agreement allows for a 50/50 share for the cost of the work that is directly performed by MSE Technology Applications. The estimated costs for the V-Tank preliminary design and laboratory study work plan are shown in Table 13. The costs have been broken out by task. The estimate is based on fully burdened rates for personnel and includes general, administrative, and overhead adds on the subcontracted costs.

Table 13. Key personnel for Test Phases 1 and 2.

Team Member Role	Team Member Name
<b>Agency Team Members</b>	
DOE WAG 1 Manager	Mark Shaw
EPA Region 10	Matt Wilkening
Idaho Department of Environmental Quality	Clyde Cody, Daryl Koch
<b>INEEL M&amp;O Contractor Team Members</b>	
WAG 1 Manager	Allen Jantz
WAG 1 Project Manager	James Jessmore
WAG 1 Project Engineer	Gary McDannel
ES-CO/S TS Principal Investigator	Eric Miller
ES-CO/S TS Lead Engineer	Dale Cresap
ES-CO/S Process Design Engineer	Richard Farnsworth
ES-CO/S Chemical Engineer	Dave Tyson
<b>MSE Technology Applications Team Members</b>	
Program Manager	Dan Battleson
Project Manager	Helen Joyce
Technical Lead	Jay Cornish
Engineering Technical Lead	Dr. Steve Kujawa
Chemistry Technical Lead	Dr. Clarence Whitworth
Test Engineers	Jody Bickford
Test Technician	Mike Brininger
Quality Assurance Officer	Rod Schwab
Safety and Health Support	Ken Reick
	Scott Nuthak
	Rick Obstar
<b>Waste Management Support</b>	
	Charles Brown
DOE = U.S. Department of Energy Idaho Operations Office	
EPA = U.S. Environmental Protection Agency	
ES-CO/S = ex situ chemical oxidation followed by stabilization	
INEEL = Idaho National Engineering and Environmental Laboratory	
M&O = management and operating	
WAG = waste area group	

Test Phase 3, the bench-scale hot study, will be performed by LRC in Lynchburg, Virginia. Some initial planning may begin in FY 2003, with the majority of the work planned for FY 2004. Table 14 shows the breakdown of these tasks. Table 15 shows the Task Phase 3 breakdown of tasks and estimated cost for the preliminary design and laboratory study.

Test Phase 4, the cold full-scale cold mockup study, will be performed at MSE Technology Applications. As with the hot bench-scale studies, some upfront work may be performed in FY 2003 with the majority of the work being performed in FY 2004. Table 14 shows the breakdown of these tasks. Table 16 shows the Phase 4 breakdown of tasks and estimated cost for the preliminary design and laboratory study.

Table 14. Breakdown of tasks and estimated cost for Test Phases 1 and 2.

Task	Cost (FY)
Annotated outline	\$11.5K (FY 2003)
Preliminary design and laboratory study work plan	\$30K
Preliminary design and laboratory study test plan	\$52.4K
Review of work plan and test plan	\$13.1K for review of test plan
Experimentation	\$199.0K
Final report	\$36.0K
Total	\$342K

FY = fiscal year

Table 15. Task Phase 3 breakdown of tasks and estimated cost for the preliminary design and laboratory study.

Task	Cost
Prepare SOW	TBD
Prepare Sampling and Analysis Plan	TBD
Preliminary design and laboratory study work plan	TBD
Preliminary design and laboratory study test plan	TBD
Review of work plan and test plan	TBD
Experimentation	TBD
Final report	TBD

SOW = Statement of Work

TBD = to be determined

Table 16. Test Phase 4 breakdown of tasks and estimated cost for the preliminary design and laboratory study.

Task	Cost
Annotated outline	TBD
Preliminary design and laboratory study work plan	TBD
Preliminary design and laboratory study test plan	TBD
Review of work plan and test plan	TBD
Experimentation	TBD
Final report	TBD

TBD = to be determined

## 16. REFERENCES

- 29 CFR 1910.120, 2003, "Hazardous Waste Operations and Emergency Response," *Code of Federal Regulations*, Office of the Federal Register, June 2003.
- 29 CFR 1910.1450, 2003, "Occupational Exposure to Hazardous Chemicals in Laboratories," *Code of Federal Regulations*, Office of the Federal Register, June 2003.
- 40 CFR 261.2, 2003, "Definition of Solid Waste," *Code of Federal Regulations*, Office of the Federal Register, June 2003.
- 40 CFR 268, 2003, "Land Disposal Restrictions," *Code of Federal Regulations*, Office of the Federal Register, January 2003.
- 40 CFR 300.440, 2003, "Procedures for Planning and Implementing Off-Site Response Actions," *Code of Federal Regulations*, Office of the Federal Register, June 2003.
- 15 USC § 2601 et seq., 1976, "The Toxic Substances Control Act (TSCA) of 1976," *United States Code*.
- 42 USC § 6901 et seq., 1976, "Resource Conservation and Recovery Act (Solid Waste Disposal Act)," *United States Code*, October 21, 1976.
- 42 USC § 9601 et seq., 1980, "Comprehensive Environmental Response, Compensation and Liability Act of 1980 (CERCLA/Superfund)," *United States Code*, December 11, 1980.
- DOE-ID, 1997, *Comprehensive Remedial Investigation/Feasibility Study for the Test Area North Operable Unit 1-10 at the Idaho National Engineering and Environmental Laboratory*, DOE/ID-10557, Revision 0, U.S. Department of Energy Idaho Operations Office, November 1997.
- DOE-ID, 2002a, *ICDF Complex Waste Acceptance Criteria*, DOE/ID-10881, Revision 0, U.S. Department of Energy Idaho Operations Office, March 2002.
- DOE-ID, 2002b, *Technology Evaluation Scope of Work for the V-Tanks, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10*, DOE/ID-10999, Revision 0, U.S. Department of Energy Idaho Operations Office, July 2002.
- DOE-ID, 2003a, *Technology Evaluation Report for the V-Tanks, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10*, DOE/ID-11038, Revision 0, U.S. Department of Energy Idaho Operations Office, April 2003.
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- EPA, 1992, *Guide for Conducting Treatability Studies under CERCLA*, Interim Final, EPA/540/2-89/058, U.S. Environmental Protection Agency.

- EPA Method 8082, 1996, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, 3<sup>rd</sup> ed., “Polychlorinated Biphenyls (PCBs) by Gas Chromatography,” Revision 0, U.S. Environmental Protection Agency.
- EPA Method 8260B, 1996, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, 3<sup>rd</sup> ed., “Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS),” Revision 2, U.S. Environmental Protection Agency.
- EPA Method 8270C, 1996, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, 3<sup>rd</sup> ed., “Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS),” Revision 3, U.S. Environmental Protection Agency.
- Form 435.28, 1998, “Solid/Hazardous Waste Determination,” Revision 2, Idaho National Engineering and Environmental Laboratory, May 1998.
- INEL, 1995a, *Data Management Plan for the Idaho National Engineering Laboratory Environmental Restoration Program*, INEL-95/0527 (formerly EGG-ERD-10227), Revision 1, Idaho National Engineering and Environmental Laboratory, June 1995.
- INEL, 1995b, *Community Relations Plan—A Guide to Public Involvement in the Environmental Restoration Program at the INEL*, Doc. Id. 8511, Revision 0, Idaho National Engineering and Environmental Laboratory, May 1995.
- INEEL, 1998, *Final Report Treatability Study for LMITCO TSF-09 V-1, V-2, and V-3 Tank Waste*, INEEL/EXT-98-00739, Revision 0, Idaho National Engineering and Environmental Laboratory, September 1998.
- INEEL, 2003a, *Cold Bench-Scale Test Plan for Chemical Oxidation/Stabilization of Surrogate V-Tank Waste (TSF-09/18) at Waste Area Group 1, Operable Unit 1-10*, INEEL/EXT-03-00860, Revision 0, Idaho National Engineering and Environmental Laboratory, July 2003.
- INEEL, 2003b, *Waste Management Plan for V-Tanks Early Remedial Action for the Test Area North, Waste Area Group 1, Operable Unit 1-10, Group 2 Sites*, INEEL/EXT-02-01608, Revision 0, Idaho National Engineering and Environmental Laboratory, May 2003.
- ITC, 1994, *Preliminary Scoping Track 2 Summary Report for the Test Area North Operable Unit 1-05: Radioactive Contamination Sites*, INEL-94/0135 (formerly EGG-ER-11162), Revision 0, Idaho National Engineering and Environmental Laboratory, October 1994.
- MCP-233, 2002, “Process for Developing, Releasing, and Distributing ER Documents (Supplemental to MCP-135 and MCP-9395),” Revision 4, *Environmental Restoration Work Processes Manual*, Idaho National Engineering and Environmental Laboratory, June 2002.
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- MCP-240, 2002, “ER/D&D Operational Review Board Process,” Revision 9, *Environmental Restoration Work Processes Manual*, Idaho National Engineering and Environmental Laboratory, November 2002.

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- MCP-1194, 2003, "Logbook Practices for ER and D&D&D Projects," Revision 1, *Environmental Restoration Work Processes Manual*, Idaho National Engineering and Environmental Laboratory, May 2003.
- MCP-3480, 2002, "Environmental Instructions for Facilities, Processes, Materials, and Equipment," Revision 8, *Manual 8–Environmental Protection and Compliance*, Idaho National Engineering and Environmental Laboratory, November 2002.
- Michael, David L., 1996, *Field Sampling Plan for Operable Unit 1-10: Test Area North*, INEL-95/0304, Revision 1, Idaho National Engineering and Environmental Laboratory, April 1996.
- PLN-476, 2002, "Records Management Plan for the Environmental Restoration Program and Projects," Revision 0, Idaho National Engineering and Environmental Laboratory, July 2002.
- PRD-5030, 2002, "Environmental Requirements for Facilities, Processes, Materials, and Equipment," Revision 2, *Manual 8–Environmental Protection and Compliance*, Idaho National Engineering and Environmental Laboratory, April 2002.



**Appendix A**  
**Agency Comments and Comment Resolutions**



# Appendix A

## DEQ Comment Resolutions-1

PROJECT DOCUMENT REVIEW RECORD				
<b>INEEL</b> <small>Institute of Nuclear Engineering &amp; Environmental Laboratory</small>				
<b>DOCUMENT TITLE/DESCRIPTION:</b> DEQ Technical Review Comments for the Laboratory Study Work Plan for Chemical Oxidation and Stabilization of V-Tanks' Contents, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10 (Draft) June 13, 2003				
<b>DATE:</b> June 18, 2003 <b>REVIEWER:</b> DER				
ITEM NUMBER	SECTION NUMBER	PAGE NUMBER	COMMENT	RESOLUTION
<b>GENERAL COMMENTS</b>				
1	NA	NA	<p>The two major oxidants discussed in this report, sodium peroxydisulfate and Fenton's Reagent, both appear to have advantages and the capability of destroying VOCs, and other organics in the V-Tanks wastes. However, if the initial Bench-Scale Cold Tests appear to strongly favor one oxidant over another, it seems that additional testing of both oxidants, involving major time and expense, could be eliminated. Available studies (see references) appear to favor peroxydisulfate for some of (but not inclusive of all) the following reasons:</p> <ul style="list-style-type: none"> <li>• Peroxydisulfate is termed in the literature as "omnivorous", meaning it is one of the strongest oxidants known and is capable of oxidizing a variety of contaminants, combined or individually. Use of peroxydisulfate may obviate the need for multiple steps in the initial bench scale testing, thought to be necessary for demonstrating the capability of peroxydisulfate to destroy the individual components of the waste (TCE, PCBs, oils, etc.)</li> </ul>	<p>Comment partially incorporated.</p> <p>Your strategy of favoring one oxidant over another, based on initial bench-scale tests, to save on additional testing, time, and expense is a good idea. The Technology Evaluation Report initially identified sodium peroxydisulfate as the chosen oxidant of choice. In the interim, a change of direction has been chosen based on processing issues. One in particular is the high total organic carbon in the sludge – present as hydraulic fluid and/or cutting oil. If it is assumed that enough sodium peroxydisulfide is needed for destruction of the entire oil phase in order to destroy the UHC organics toward their treatment standards, the density of the resulting solution becomes so high that grouting becomes difficult unless a great deal of makeup water is used. The current strategy in the Conceptual Design Report has us using a combination of hydrogen peroxide and sodium peroxydisulfate. The MSE Test Plan will however will use one oxidant (i.e., hydrogen peroxide) during treatment. Sodium peroxydisulfate is only reserved for testing if peroxide is unsuccessful. This strategy is captured in MSEs planning documentation. <b>A footnote is placed into the 1<sup>st</sup> test objective to inform the reader that the test protocol for the oxidant selection is to favor Fenton's reagent.</b></p>

## PROJECT DOCUMENT REVIEW RECORD

**DOCUMENT TITLE/DESCRIPTION:** DEQ Technical Review Comments for the Laboratory Study Work Plan for Chemical Oxidation and Stabilization of V-Tanks' Contents, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10 (Draft) June 13, 2003

**DATE:** June 18, 2003    **REVIEWER:** DER

ITEM NUMBER	SECTION NUMBER	PAGE NUMBER	COMMENT	RESOLUTION
1	Continued		<ul style="list-style-type: none"> <li>The process operates below 100 C, thus minimizing offgas volume and reducing or eliminating the formation of dioxins and furans in the offgas and reducing offgas treatment needs. This appears to be contrary to information presented on Page 12 of the document (see General Comment 9). A taken to eliminate concern over volatilizing VOCs.</li> <li>Oxidation using peroxydisulfate in an alkaline solution has been found to avoid the formation and release of chlorine from organic molecules from chlorine-containing wastes. The chlorine released from the destruction process remains as chloride ion in solution. This would facilitate less wastes to be treated in an off-gas system.</li> <li>Peroxydisulfate apparently can be recycled, through electrolysis. This should be looked at to see if recovery and recycling of the reagent would be cost effective.</li> </ul>	<p>In Section 4.1 is the passage:</p> <p>“From the above list of test objectives is the flexibility to find a set of less aggressive test conditions that adequately treats the waste in a suitable reaction time. If these less aggressive test conditions are found, further testing at more aggressive bounding conditions would not be necessary. This type of flexibility allows the laboratory to forgo unnecessary testing.”</p> <p>This passage is the part of the document that allows us to exercise some freedom to avoid testing that, in time, may become unnecessary. This will help the study to stay on schedule and work towards the a viable process for treatment and scale-up.</p>

## PROJECT DOCUMENT REVIEW RECORD

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DATE: June 18, 2003		REVIEWER: DER		
ITEM NUMBER	SECTION NUMBER	PAGE NUMBER	COMMENT	RESOLUTION
SPECIFIC COMMENTS				
1	1.3.1, first paragraph	3	This paragraph (last sentence) states that resin beads and ceramic-like particles were found in the (1980) samples. These materials have never really been described to our knowledge, nor their origin. Please discuss, and also describe any implications for the bench-scale and full-scale tests, and remedial action, if any.	Commented noted but not incorporated.  The information on the resin beads was taken from historical data. There was no explanation in that reference for why they were present. These resins have not been reported in subsequent sampling in '93/96. Other chemical oxidation systems (e.g., AEA) have successfully oxidized organic resins. There should be no implications for our studies or remedial action.
2	1.3.1, fourth paragraph	3	This paragraph describes the “two separate liquid sampling campaigns” of the V-tanks conducted in 1990. Are there results available for these sampling events, and why are they not listed similar to the 1980, 1993 and 1996 sampling events?	Comment incorporated.  There are results for this sampling campaign, but it was decided to use the 1993/1996 data because the 1990 data only looked for RCRA characteristic metals, VOCs, and SVOCs (8 metals, 10 VOCs, and 14 SVOCs) while the other campaigns analyzed for much more than these. This will be added to text.
3	1.4, Table 4	7	This table lists the radionuclides in the V-Tanks’ contents. Please add radiological measurement units (i.e., “pCi/g”) to this table for these wastes, and break down the “Transuranic waste” listing to show isotopes, if available. Also, indicate in the table heading, or a footnote, that these analyses reflect the combined liquid and sludges found in the V-Tanks.	Comment Incorporated.  The units of nCi/g were added and two footnotes were added: one to indicate that the V-tank information was based on the combined liquid and sludge phases and one to indicate the exact TRU isotopes that make up the overall source term.
4	1.6, Item 2a, 1-4	9	This list appears to be missing the second item of the Test Plan, or the numbering is off. Please correct.	Comment incorporated.  This numbering is fixed.

## PROJECT DOCUMENT REVIEW RECORD

**DOCUMENT TITLE/DESCRIPTION:** DEQ Technical Review Comments for the Laboratory Study Work Plan for Chemical Oxidation and Stabilization of V-Tanks' Contents, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10 (Draft) June 13, 2003

DATE: June 18, 2003 REVIEWER: DER			
ITEM NUMBER	SECTION NUMBER	PAGE NUMBER	RESOLUTION
5	2.1, Table 6	11	<p>The last column in this table has a heading of "Without 6000 (%)." Please clarify the meaning of this heading in a footnote or in the text.</p> <p>Comment incorporated.</p> <p>The heading has been changed to "Destruction Efficiency without 6000 gallons." A footnote has been added to indicate that 6000 gallons of liquid supernate is removed from Tank V-3 in this scenario.</p>
6	2.1, Bullet 2	11	<p>This list states the assumptions for the listed destruction efficiencies. Bullet 2 states that no credit is taken for the mass of oxidants used. Please state whether this assumption is also valid for actual waste treatment efficiencies.</p> <p>Comment incorporated.</p> <p>The following footnote was added: "These destruction efficiencies represent the worst-case. Credit for oxidant weight increase and stabilization additives will be used to establish achievement of final UTS values."</p>
7	2.1, Bullet 2	11	<p>Bullet 3 states that no credit is taken for the mass of additives used to produce the final cemented waste form. Is this assumption also valid for actual waste treatment efficiencies?</p> <p>Comment incorporated.</p> <p>The following footnote was added: "These destruction efficiencies represent the worst-case. Credit for oxidant weight increase and stabilization additives will be used to establish achievement of final UTS values."</p>
8	2.2, paragraph 1	12	<p>This paragraph states that sodium peroxydisulfate and Fenton's reagent will be studied as options. Please state whether the use of one of these oxidants results in a significant reduction in treated waste volume, compared to the other.</p> <p>Comment noted but not incorporated</p> <p>Persulfate may result in lower waste volume but due to density increase and potential grouting issues, peroxide appears to be the preferred oxidant for proven effective.</p>

## PROJECT DOCUMENT REVIEW RECORD

**DOCUMENT TITLE/DESCRIPTION:** DEQ Technical Review Comments for the Laboratory Study Work Plan for Chemical Oxidation and Stabilization of V-Tanks' Contents, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10 (Draft) June 13, 2003

DATE: June 18, 2003    REVIEWER: DER			
ITEM NUMBER	SECTION NUMBER	PAGE NUMBER	COMMENT
9	2.2, paragraph 2	12	<p>It is not clear, based on this discussion, why peroxydisulfate would be considered in light of the fact that the elevated temperatures apparently conducive to the use of this oxidant "may volatilize significant portions of VOCs from the oxidant bath." The attractiveness of oxidation as a remedy has been in part based on the fact that oxidation would destroy the VOCs insitu, and therefore not require capture and eventual treatment of captured VOCs. Please discuss why peroxydisulphate would be considered if it appears that higher temperatures will be required that may result in the release of untreated VOCs.</p>
			<p><b>RESOLUTION</b></p> <p>Comment noted but not incorporated</p> <p>Both oxidant systems have the capability to release VOCs. In addition, the temperature of the hydrogen peroxide oxidation may be allowed to elevate if safety concerns dictate. For instance, AEA Technologies is employing a 100°C hydrogen peroxide destruction of resin (at ORNL). The higher temperature ensures that a build-up of peroxide radicals does not cause an uncontrolled expulsion associated with rapid gas generation due to run-away reactions.</p> <p>In our Conceptual Design Report, the temperature is initially 40°C with peroxide to destroy the VOCs. VOCs are thought of as easier to destroy and at 40°C, little volatilization out of system should occur. After the VOCs are destroyed, then the temperature can be raised to attack the less volatile species.</p>
10	3, first bullet, items 1, 6	14	<p>Both describe processes that appear redundant. Consider deleting one unless there is a reason to retain both.</p>
			<p>Comment incorporated.</p> <p>6th bullet was deleted.</p>

## PROJECT DOCUMENT REVIEW RECORD

**DOCUMENT TITLE/DESCRIPTION:** DEQ Technical Review Comments for the Laboratory Study Work Plan for Chemical Oxidation and Stabilization of V-Tanks' Contents, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10 (Draft) June 13, 2003

DATE: June 18, 2003 REVIEWER: DER			
ITEM NUMBER	SECTION NUMBER	PAGE NUMBER	
			COMMENT
			RESOLUTION
11	Table 7	16	<p>Please explain why, for the objective "Determine effectiveness of Chemical Oxidation," TCLP: Method 1311 is considered necessary. It seems TCLP should only be necessary for the next objective, "Determine effectiveness of stabilization agent.t."</p>
			<p>Comment incorporated.</p> <p>The following passage has been added:</p> <p>"Measurement of this change, via the TCLP test, determines potential differences in the leachability of toxic metals due to the oxidation process. Possible changes may occur due to alterations of metallic valence states/speciation. This measurement will provide an indication for the level of metal fixation required for the stabilization step. In practice, TCLP analysis for metals would not be mandatory prior to stabilization. As an experimental measure, useful information may be found."</p>
12	Table 7	17	<p>It is not clear what is meant by "V-Tank sample" in the "Objective" column. Please consider expanding on the objective and add a verb similar to the other objectives (for example, "Testing"). Also, it is also not clear why "TCLP: Method 1311" appears in the Proposed Method column for this objective. Please explain.</p>
			<p>Comment incorporated</p> <p>This is a formatting issue for the table. The table entry did not "wrap the text". The front part of the objective is at the bottom of page 16. Text editing will fix this error.</p>
13	4.1.1, 1a (3) and (4)	19	<p>It is not clear what "persulphate" is. There is not an accompanying discussion in the text. Please explain. Also, it is not clear what the difference is between process elements 3 and 4, besides the reversal of the two elements (oxidants).</p>
			<p>Comment Incorporated.</p> <p>The oxidant in actuality should be referred to as persulfate. The document will be searched for the term peroxydisulfate and replace with persulfate.</p>

## PROJECT DOCUMENT REVIEW RECORD

**DOCUMENT TITLE/DESCRIPTION:** DEQ Technical Review Comments for the Laboratory Study Work Plan for Chemical Oxidation and Stabilization of V-Tanks' Contents, TSF-09/18, at Waste Area Group 1, Operable Unit 1-10 (Draft) June 13, 2003

DATE: June 18, 2003 REVIEWER: DER				
ITEM NUMBER	SECTION NUMBER	PAGE NUMBER	COMMENT	RESOLUTION
14	4.2, first paragraph	23	It is suggested the next to last sentence would be more clear if it deleted "Due to the presence of VOCs in the waste" and started with "There is expected to be depleted (a lab term?) amount that will require the subcontacted laboratory to spike the samples to levels similar to levels that will be found in the actual mixed V-tank wastes."	Comment Incorporated.  This sentence will be altered as requested. In addition, a brief passage will be added to describing a sampling effort in FY04 that will piggy-back a tank consolidation effort that is described in the conceptual design report.
15	5.1.3, second bullet	24	The oxidants include "sodium persulphate." It appears this term is being used interchangeably with "sodium peroxydisulphate." It is not clear if this is ever explained in an earlier section of the document.	Comment Incorporated.  The document will be searched for the term peroxydisulfate and replace with persulfate.

**PROJECT DOCUMENT REVIEW RECORD**

**DOCUMENT TITLE/DESCRIPTION:** EPA Comments on Laboratory Study Work Plan for Chemical Oxidation and Stabilization of V-Tanks' Contents  
TSF-09/18, WAG 1, OU 1-10

**DATE:** June 18, 2003 **REVIEWER:** Dave Tyson

ITEM NUMBER	SECTION NUMBER	PAGE NUMBER	COMMENT	RESOLUTION
<b>GENERAL COMMENTS</b>				
1	NA	NA	<p>The Work Plan is somewhat unclear and difficult to follow. Part of the lack of clarity arises from an abrupt introduction of "surrogates" on page 6. No background is presented leading to the need for, and definition of, the "surrogates" in the present context. The Work Plan does not provide enough information to establish the value of the Test Phase 1 (Bench-scale cold tests screening of parameters) and Test Phase 2 (Bench-scale cold tests validation of accepted parameters) to justify a total expense of \$342,000 for a bench scale test for an ultimate remediation of 11,912 gals (about 60 cu. yds.) of wastes. While EPA recognizes that the presence of radioactive materials warrants special attention from a health and safety point of view this cost seems excessive. EPA's contractor, Gannett Fleming, reviewed projects involving non-radioactive waste but characterized with all the other constituents in the V-tank wastes, found that a final remedial cost of \$100 to \$300/cu. yd. would be considered typical.</p>	<p>Comment incorporated</p> <p>The following sentence will introduce surrogates in Section 1.4 "Due to the extensive testing planned, When surrogate feedstocks will be used in the preliminary laboratory testing. The surrogate feedstock will allow for screening and refining of the treatment procedure. Once completed, actual tank waste will be used for testing."</p> <p>Prior to starting right out on actual wastes, many treatability studies utilize surrogates to fine-tune the treatment process of interest. Due to limited amount of available hot material (and large expense and exposure to get more) refining the operating conditions through surrogate testing will be more cost effective. Due to the physical and chemical nature of the V-tank waste, uncertainties exist for the implementation of the CO/S process. A preliminary study last summer at MSE showed difficulty in destroying hexachlorobenzene (mixture of hexachlorobenzene/biphenyl was used as a low cost substituted for Aroclor-1260). CO/S has been studied in-depth for destroying organics in the aqueous phase. Our data indicates that the bulk of the organics reside in the sludge phase – present as hydraulic/cutting oil per the analytical data and process knowledge. As a result, the destruction will have to be in the "non-aqueous" phase and we need data to support that this will not be a problem.</p>

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1	Continued		Instead a hot bench scale test, followed by a full scale hot mockup test appears to be more adequate and cost-effective.	<p>Furthermore, we will need to achieve UTS standards for the organics. Table 6 of the Work Plan shows the theoretical DREs and final organic concentration that will be needed. Obviously, chemical oxidation will achieve a fair amount of organic destruction, but we need to satisfy ourselves that these low limits for the UTS organics can be achieved.</p> <p>The previous treatability study on hot waste performed for the INEEL - using CO/S - back in 1997/1998 did not examine bis (2-ethylhexyl) phthalate destruction and there currently is no literature on the DRE for this constituent using chemical oxidation. The DREs reported for PCE, TCE, and PCB may have been biased high due to a lack of material balance closure, i.e. they may have lost much VOCs due to mixing in an open vessel/beaker. Another key reason to do the cold testing now is to get this testing done prior to completion of the ROD amendment. (This would not be possible with actual V-Tank waste due to the logistics of getting this material to the lab for testing and the limited quantity.) In the event the MSE testing fails to validate the BWXT study, it may be necessary to write a contingent ROD. If pursued, however, stabilization alone would not be considered for the reasons provided in the TER and comparable comments addressed at that point.</p>

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1	Continue			<p>The cost of the cold testing is probably not near the percentage that is reported in this comment. The cost to treat a few gallons is almost as great as the cost to treat millions of gallons. The reason for this is that the up-front work is essentially equivalent regardless of the quantity to be treated. It still takes a safety analysis, it still requires a design, it still requires operating procedures, etc. The actual cost during the operation is but a fraction of the total, particularly in our case when the quantity to treat is so small.</p>

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2	NA	NA	<p>Some changes in the Work Plan are noted from what was described in the selection of ES-CO/S in earlier reports that were reviewed. In those earlier reports, INEEL proposed to raise the pH within the Glass Lined Reaction Vessel (GLRV) to 12 by the addition of 50wt% sodium hydroxide. It appears this step is not included in the Work Plan (other than the mentioning the word caustic in the 3rd bullet in Section 5.1.3 and the 3rd bullet in Section 5.2.3). On the contrary, on page 12, the text states that</p> <ol style="list-style-type: none"> <li>1. For oxidant #1, hydrogen peroxide (Fenton's Reagent), the system works best in a pH range of 3 to 5.</li> <li>2. For oxidant #2, peroxydisulfate, the system works best at pH 2 to 3.</li> </ol> <p>No explanation is provided why the step of adding 50wt% sodium hydroxide was eliminated in the Work Plan. Further, no explanation was given how the pH of the waste (pH 8.6-9.57) will be lowered to the ranges 3 to 5 and 2 to 3.</p> <p>In contrast to the original conceptual design of raising the pH to 12 as described in the previous reports, the planned lowering of pH to 2 to 5 in the Work Plan is expected to release metals. It is likely that this release of metals in solution will reduce the efficacy of the subsequent step of stabilization.</p>
			RESOLUTION
			<p>Comment Incorporated.</p> <p>The following sentence was added to Section 2.3: "The stabilization step requires that the post-oxidant slurry have a basic pH. The slurry will have to be adjusted from an acidic pH to a basic pH. Sodium hydroxide can be used to raise the slurry."</p> <p>The lower pHs reported are only to facilitate the reaction kinetics. The rate limiting step for the oxidation kinetics is the formation of free radicals from either peroxide or peroxydisulfate. Particularly in the case of peroxide, operation outside of a low pH will slow or hinder radical formation – thus, preventing any organic destruction from occurring. After oxidation is completed, the pH has to be raised using a strong base (NaOH) in order to meet the necessary pH range required for stabilization (i.e. fairly basic). The bottom line is that we are not concerned with metals mobility in the first step (oxidation) – we are only concerned with organic destruction. In the second step (stabilization), where we are greatly concerned with metals mobility, we are raising the pH to the basic regime. As a side note, we are planning to perform a TCLP on the oxidized product to quantify the extent of metal dissolution that occurs – mostly to give us an indication of what is exactly happening.</p>

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2	Continued		Once again, this revives the earlier comment that stabilization alone without oxidation may be an appropriate alternative. Oxidation is needed primarily to remove the VOCs and SVOCs which are generally within the range amenable for stabilization alone. Tank V-9 has a higher concentration of TCE relative to the other three (3) tanks. However, V-9 has only 320 gals out of a total of 11,912 gals, and since the wastes from all the tanks will be mixed prior to treatment, the average TCE concentration of the waste to be treated would be significantly lower. Hence, dissolution of metals by lowering pH as now proposed in the Work Plan raises further questions about the value of the oxidation step. In conclusion, it is still prudent to evaluate the stabilization alone option.
3	NA	NA	<p>If INEEL still decides to pursue ES-OC/S, then a hot bench scale test followed by a hot mockup test appears to be adequate. In particular, since there is a likelihood of difference in optimum parameters for the cold test (Phase 2) and hot test (Phase 3), the value of the cold mockup test is questionable. Even if INEEL wishes to perform bench tests for ES-OC/S, it would be of value to perform a separate bench test for the stabilization alone option and evaluate if the additional resources devoted to the oxidation step is justifiable.</p> <p>Comment noted but not incorporated.</p> <p>See the answer to Comment 1 (regarding the need for surrogate tests).</p> <p>See the answer to Comment 2 (regarding the need for stabilization alone tests).</p>

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4	NA	NA	<p>In general, Table 7 is unclear. Perhaps a flow chart with sampling nodes identified will make it clearer.</p> <p>Additionally, Table 7 would be clearer with the Test Phase numbers and titles identified as row headings. On Table 7, it appears there are a number of repetitive tests. For example VOC, SVOC and PCB tests are indicated for both post-oxidation and pre-stabilization, even though these refer to the same point in the sequence of activities. As an additional example, it is not clear why it is necessary to perform the TCLP test in the intermediate (post oxidation) step. One test before oxidation and one after stabilization should be sufficient. On Table 7, several entries shows "TBD". Presuming that it stands for "To be determined," some explanation should be provided as to what is holding up the explicit selection. The assignment of numerous TBDs clouded the issue of "screening." It is not clear what "screening" stands for in the context of the Work Plan.</p>	<p>Comment Incorporated</p> <ul style="list-style-type: none"> <li>The format of Table 7 will be altered to include the Test Phase numbers and titles.</li> <li>As far as post-oxidizing TCLP tests are concerned, we are using the surrogates to learn as much as we can about the process. The information gathered in the post-oxidizing TCLP tests will help us determine if the chemical oxidizing step, in some ways, alters the speciation of the UTS metals to render them more soluble some of the UTS metals. New text has been added to clarify this point.</li> <li>The TBDs are designed to provide MSE with the flexibility to determine their testing protocol. ICP has the right to review testing, procedures, and provide oversight. ICP is also reviewing, amending, and reviewing the test plan. Screening refers to testing to evaluate one thing against another, i.e. is one oxidant better than another, does one temperature promote reaction better than another, etc. The following text has been added Section 1.6: "Screening tests produce data that are taken for preliminary selection or preliminary adjustment of mix parameters."</li> </ul>

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<b>SPECIFIC COMMENTS</b>				
1	Line 3	2	"...wastes in all tanks was removed in 1982. The tanks have not been used since the 1980's..." If wastes were removed in 1982, then what does the wastes on Table 1 refer to?	Comment Incorporated:  This sentence will be removed--the referenced report used for this information is not clear on this issue.
2	Table 5	10	It is not clear how the grain size distribution is proposed to be used.	Comment noted but not Incorporated.  Grain size information provides a background on the waste and has no impact on surrogate formulation.
3		14	1st and the 6th sub-bullet of the 1st bullet: Both bullets appear to be the same.	Comment Incorporated:  The 6th bullet will be removed.
4	Last 6 lines	19	Preceding text described the use of peroxydisulfate and Fenton's Reagent. However, there was no indication in that text that two other oxidation sequences will be tried using mixtures of the two oxidants.	Comment Incorporated  The following text has been added to Section 2.2:  "In addition to using a single oxidant to treat the contaminants and the resident oily phase, a combination of the two oxidants can be used."
5	Heading 8a	20	It is not clear what "nominal" implies.	Comment Incorporated  "Nominal" will be deleted and replaced by "Average composition of V-tank contents"
6	Objective 2	21	Is INEEL proposing to take measurements at intermediate points in time in addition to the measurements at the two	Comment Incorporated.

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			extremes? If so, the objective of the measurements at intermediate points in time was not discussed.	<p>The following text was added: "In order to determine kinetic rates, periodic sampling or on-line monitoring (e.g., pH, temperature, etc.) needs to be implemented so that the changes with respect to time can be plotted. This would allow the chemical process to be better understood and provide good estimates for reaction times needed to achieve required extent of reactions."</p> <p>In Objective 2, we discuss obtaining kinetic data. Kinetic data can only be obtained through periodic sampling so that concentrations can be plotted versus time. This allows us to understand the interplay of the rate-limiting step (radical formation – 1st order kinetics that is governed by oxidant input rate) with the rapid-rate step of organic destruction (2nd order kinetics governed by organic contaminant concentration). So essentially we need measurements at intermediate points.</p> <p>Purely from a regulatory compliance stand point, sampling at the extremes (beginning and end of stabilization) is enough. However, to understand the chemistry, we need to sample at intermediate steps. Although I think this is tied more to TO #10, we want to see if the oxidant(s) selectively destroy the hazardous constituents as some literature indicates. The only way to do this is through sampling at intermediate steps. This has the potential to significantly reduce the required amount of oxidant (orders of magnitude) which in turn reduces the amount of grout required - all of which will reduce the final remediation cost.</p>

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				They may actually have a point with some of the things we are looking for at these intermediate steps. Your point about oxidation affecting stabilization/leachability seems valid. We certainly want to see what the effects are on the organics at these intermediate steps. However, the need to test things like durability may not be necessary.
7	TO 5	21	Since INEEL is only interested in improvement in durability from the initial state to the final state after stabilization, the value of durability determination at the intermediate step of post-oxidation was not discussed and is questionable.	<p>Comment Incorporated.</p> <p>This text was added to Test Objective 5: "Testing for this objective will involve the use of stainless steel coupons in the oxidation bath and measuring for pitting and corrosion."</p> <p>This Test Objective addresses the piping and equipment that will receive the post-oxidized sludge which is initially acidic and high in free chlorides in solution (fairly corrosive sludge). We want to ensure that our equipment will hold up in the short term for neutralization (slightly basic) and stabilization. Tests affiliated with this objective will be testing with steel coupons and measuring corrosion/pitting.</p>

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8	TO 6	21	Since INEEL is only interested in reduction in leachability from the initial state to the final state after stabilization, the value of the estimation of the changes in leachability at the intermediate step of post-oxidation was not discussed and is questionable.	Comment incorporated.  The TCLP test will only be performed after oxidation is completed.  The following text was added: "Measurement of this change, via the TCLP test, determines potential differences in the leachability of toxic metals due to the oxidation process. Possible changes may occur due to alterations of metallic valence states/speciation. This measurement will provide an indication for the level of metal fixation required for the stabilization step. In practice, TCLP analysis for metals would not be mandatory prior to stabilization. As an experimental measure, useful information may be found."
9	Last Paragraph	22	It appears that INEEL is prepared to deal with a substantially different response with the hot sample than from the cold one. Hence the question arises about the need for Test Phases 1 & 2.	Comment noted but not Incorporated  See comment to General Comment #1
10	5.1.3	24	Is simulated waste different from surrogate? If the answer is affirmative, than the difference should be explained.	Comment Incorporated  The word "surrogate" was added.

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11	5.2.3	23	Even though the addition of sodium hydroxide was not discussed earlier in the report it appears here. This in conjunction of the fact that the Work Plan recommends lowering the pH to 2-5, renders the Work Plan confusing.
			Comment Incorporated. The following text is added to Section 2.3. "The stabilization step requires that the post-oxidant slurry have a basic pH. The slurry will have to be adjusted from an acidic pH to a basic pH. Sodium hydroxide can be used to raise the slurry."
12	Table 9	27	The distinction between the entries in column 4 and those in columns 2 & 3 is not clear and should be clarified.
			Comment Incorporated. The following text was added: "The supernatant liquid and the sludge are combined within the same sample bottle."
13	Section 8.1	29	Note that EPA no longer uses the concept of Level I through Level V data. The terms used now are Screening Data with Definite Confirmation and Definite Data. The reference for this is the 1993 EPA document Data Quality Objective Process for Superfund, September 1993, OSWER publication 9355.9-01. The text should be changed to reflect this terminology.
			Comment Incorporated. The text was changed to replace the old terms with these new terms.

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14	2 <sup>nd</sup> paragraph	33	As shown in Table 4, the highest concentration of PCBs in Tank V-9 is 95.9 mg/kg. 40CFR 761.61 now allows 100 mg/kg to be left on site with appropriate engineering controls. Since the 320 gals of V-9 wastes will be mixed with contents of other tanks containing 11,592 gals (with a highest PCB concentration of 34.6 mg/kg in Tank V-1 as shown in Table 4), the concentration in the mixed waste stream will be substantially lower than 95.9 mg/kg. Further, since the wastes will be stabilized with relatively larger mass of cement and other substances, the concentration of PCBs in the stabilized waste will significantly lower than 95.9 mg/kg. Additionally, the stabilized waste will be disposed in ICDF. Hence, TSCA should not be an issue.	Comment noted but not Incorporated.  As with RCRA, TSCA regulations are applicable at the point at which the waste first becomes regulated. TSCA regulations require the characterization and analysis of multiphasic solutions in separate phases, liquid and dry, 40 CFR 761.1(b)(4). When the composite V-Tank waste is characterized for PCB concentration in the dry solids, the concentration is approximately 294 ppm. Because we have no intent to separate the phases, TSCA regulations require that we manage the joint waste stream at the concentration of the highest phase. That is well above the allowance for leaving PCB remediation waste in place.  The allowance for 100 mg/kg to be left on site 761.61(a)(4)(i)(B)(3) is applicable only when waste is left in place. That is not an option for the V-tank waste because of the other contamination present such as the radionuclides and other RCRA organics. Once the waste is removed from the tanks, this option is no longer applicable. This allowance is also only applicable to non-liquid PCB remediation waste 761.61(a)(4)(i). The cleanup level for liquids under this section 761.61(a)(4)(iv) refers you to the decon standards which would require you to get below 0.5 ppb. This approach was not considered further.
15	Lines 7 & 8	38	The distinction between the roles of the Project Engineer and Process Engineer is not clear since the same task heading "Conceptual Design" is assigned to both on Figure 3.	Comment Incorporated.  The wording was changed to delineate the two positions.

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16	Lines 11 to 14	8	It is unclear why monthly reports should discuss the work performed in the former week only.	Comment Incorporated. References to weeks are replaced and months are added.
17	Table 12	40	Even though preparation for the Work Plans for the various test phases are shown on Figure 2 (e.g., PDSWP), they are not identified on Table 12. Also, it is not clear what "Secondary" and "NA" on this table stand for	Comment Incorporated. Text and table will be altered to match up better. A sentence will be added to define these terms.
18	Line 6	12	The text that reads as "radical scan" should read as "radicals can".	Comment Incorporated This change has been made.
19	Line 3	28	"(INEL 1995)" should read as "(INEL 1995a)".	Comment Incorporated This change has been made.
20	7 <sup>th</sup> line from bottom	33	"INEEL" should read as "INEEL".	Comment Incorporated This change has been made.